Exhibit AA

INTERNATIONAL STANDARD

ISO 22262-1

First edition
2012-07-01

Air quality — Bulk materials —

Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials

Qualité de l'air — Matériaux solides —

Partie 1: Échantillonnage et dosage qualitatif de l'amiante dans les matériaux solides d'origine commerciale

Reference number
ISO 22262-1:2012(E)

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Published in Switzerland

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22262-1 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

ISO 22262 consists of the following parts, under the general title *Air quality — Bulk materials*:

— *Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials*

The following part is under preparation:

— *Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods*

Introduction

In the past, asbestos was used in a wide range of products. Three varieties of asbestos found extensive commercial application. Chrysotile accounted for approximately 95 % of consumption, and this variety is therefore likely to be encountered most frequently during the analysis of samples. Materials containing high proportions of chrysotile asbestos were used in buildings and in industry for fireproofing, thermal insulation, and acoustic insulation. Chrysotile was also used to reinforce materials to improve fracture and bending characteristics. A large proportion of the chrysotile produced was used in asbestos–cement products. These include flat sheets, tiles and corrugated sheets for roofing, pipes and open troughs for the collection of rainwater, as well as pressure pipes for supply of potable water. Chrysotile was also incorporated into products such as decorative coatings and plasters, glues, sealants and resins, floor tiles, gaskets, and road paving. In some products, chrysotile was incorporated to modify rheological properties, e.g. in the manufacture of ceiling tile panels and oil drilling muds. Long textile grade chrysotile fibre was also used to manufacture woven, spun, felted and paper products.

Amosite and crocidolite accounted for almost all of the remaining asbestos use. Amosite was widely used as fireproofing and in thermal insulation products, e.g. pipe coverings and insulating boards. Crocidolite was also used as fireproofing and in thermal insulation products, but was particularly prized because it is highly resistant to acids, flexible enough to be spun and has high tensile strength for reinforcement. Crocidolite found application as a reinforcing fibre in acid containers such as those used for lead–acid batteries, in high-performance textiles and gaskets, and was particularly important for the manufacture of high-pressure asbestos cement pipes for delivery of potable water.

Three other types of asbestos are currently regulated. Materials containing commercial anthophyllite are relatively rare, but they have also been used as a filler and reinforcing fibre in composite materials, and as a filtration medium. Tremolite asbestos and actinolite asbestos were not extensively used commercially, but some occurrences of tremolite asbestos in surfacing materials and fireproofing have been found in Japan. Tremolite asbestos and actinolite asbestos sometimes occur as contaminants of other commercial minerals. Other minerals can also occur as asbestos. For example, richterite asbestos and winchite asbestos occur at mass fractions between 0,1 % and 6 % associated with vermiculite, formerly mined at Libby, Montana, USA. Vermiculite from this source was widely distributed and is often found as loose fill insulation and as a constituent in a range of construction materials and fireproofing.

While the asbestos mass fraction in some products can be very high and in some cases approach 100 %, in other products the mass fractions of asbestos used were significantly lower and often between 1 % and 15 %. In some ceiling tile panels, the mass fraction of asbestos used was close to 1 %. There are only a few known materials in which the asbestos mass fraction used was less than 1 %. Some adhesives, sealing compounds and fillers were manufactured in which asbestos mass fractions were lower than 1 %. There are no known materials in which asbestos was intentionally added at mass fractions lower than 0,1 %.

In this part of ISO 22262, procedures for collection of samples and qualitative analysis of commercial bulk materials for the presence of asbestos are specified. The primary method used to identify asbestos is polarized light microscopy. Because of the wide range of matrix materials into which asbestos was incorporated, polarized light microscopy cannot provide reliable analysis of all types of asbestos-containing materials in untreated samples. The applicability of polarized light microscopy can be extended by the use of simple treatments such as ashing and treatment with acid. Optionally, either scanning electron microscopy or transmission electron microscopy may be used as an alternative or confirmatory method to identify asbestos.

Although this part of ISO 22262 specifies that, optionally, a visual estimate of the asbestos mass fraction within very broad ranges may also be made, it is recognized that the accuracy and reproducibility of such estimates is very limited. Quantitative determination of the asbestos content can be needed for a number of reasons, e.g. assessment and management of the risk from asbestos materials in buildings or to comply with regulatory definitions for asbestos-containing materials. The necessity to quantify asbestos in a material depends on the maximum mass fraction that has been adopted by the jurisdiction to define an asbestos-containing material for the purpose of regulation. Definitions range from “any asbestos” to 0,1 %, 0,5 % or 1 %. For jurisdictions in which an asbestos-containing material is defined as one containing “any asbestos”, a particular problem is how to determine whether a material does not contain asbestos, since all methods have limits of detection.

For practical purposes, since no known commercial materials exist in which commercial asbestos was intentionally added at mass fractions lower than 0,1 %, this part of ISO 22262 specifies that samples be classified as asbestos-containing (i.e. containing more than 0,1 % asbestos) if either chrysotile, amosite, crocidolite or anthophyllite, or any of these varieties in combination, is detected in the analysis. When the definition of an asbestos-containing material is either 0,5 % or 1 %, depending on the nature of the product, it is often necessary to proceed to other parts of this International Standard in order to quantify the asbestos for the purpose of defining the regulatory status of the material.

The occurrence of tremolite, actinolite or richterite/winchite in a material is usually a consequence of natural contamination of the constituents, and the detection of these minerals does not necessarily indicate that the mass fraction is more than 0,1 % asbestos. Accordingly, determination of the regulatory status of these materials by any of the criteria can often be achieved only by quantitative analysis. Since these minerals were not specifically mined and utilized for their fibrous properties, they may also occur in materials as either non-asbestiform or asbestiform analogues, or as mixtures of both. Evaluation of these types of material may require a more detailed analysis.

Simple analytical procedures such as polarized light microscopy are not capable of detecting or reliably identifying asbestos in some types of commercial products containing asbestos, either because the fibres are below the resolution of optical microscopy or because the matrix material adheres too strongly to the fibres. For these types of product, it may be necessary to utilize electron microscopy.

For a list of parts of this International Standard, see the Foreword.

The method specified in this part of ISO 22262 is based on MDHS 77,[11] VDI 3866 Part 1,[13] VDI 3866 Part 4,[14], VDI 3866 Part 5,[15], AS 4964-2004,[8] EPA/600/R-93/116,[10] and NF X46-020:2008.[12]

Air quality — Bulk materials — Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This part of ISO 22262 specifies methods for sampling bulk materials and identification of asbestos in commercial bulk materials. This part of ISO 22262 specifies appropriate sample preparation procedures and describes in detail the procedure for identification of asbestos by polarized light microscopy and dispersion staining.

This part of ISO 22262 also specifies simple procedures for separation of asbestos fibres from matrix materials such as asphalt, cement, and plastics products. Optionally, identification of asbestos can be carried out using scanning electron microscopy or transmission electron microscopy with energy dispersive X-ray analysis. Information is also provided on common analytical problems, interferences and other types of fibre that may be encountered in the analysis.

This part of ISO 22262 is applicable to qualitative identification of asbestos in specific types of manufactured asbestos-containing products and commercial minerals. This part of ISO 22262 is applicable to the analysis of fireproofing, thermal insulation, and other manufactured products or minerals in which asbestos fibres can readily be separated from matrix materials for identification.

NOTE This part of ISO 22262 is intended for use by microscopists who are familiar with polarized light microscopy methods and the other analytical procedures specified (References [16]–[19]). It is not the intention of this part of ISO 22262 to provide instruction in the fundamental analytical techniques.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
achromat
microscope objective in which chromatic aberration is corrected for two wavelengths and spherical aberration and other aperture-dependent defects are minimized for one other wavelength (usually about 550 nm)

EXAMPLE One wavelength less than about 500 nm, the other greater than about 600 nm.

NOTE This term does not imply any degree of correction for curvature of image field; coma and astigmatism are minimized for wavelengths within the achromatic range.

[ISO 10934-1:2002,[3] 2.6]

2.2
acicular
shape shown by an extremely slender crystal with cross-sectional dimensions which are small relative to its length, i.e. needle-like

[ISO 13794:1999,[4] 2.1]

2.3
alpha refractive index
α
lowest refractive index exhibited by a fibre

2.4
amphibole
group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, and having the nominal formula:

$$A_{0-1}B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where

A is K, Na

B is Fe^{2+}, Mn, Mg, Ca, Na

C is Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+}

T is Si, Al, Cr, Fe^{3+}, Ti

NOTE In some varieties of amphibole, these elements can be partially substituted by Li, Pb, or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon:oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124°.

[ISO 13794:1999,[4] 2.2]

2.5
amphibole asbestos
amphibole in an asbestiform habit

[ISO 13794:1999,[4] 2.3]

2.6
analyser
polar used after the object to determine optical effects produced by the object on the light, polarized or otherwise, with which it is illuminated

NOTE It is usually positioned between the objective and the primary image plane.

[ISO 10934-1:2002,[3] 2.117.1]

2.7
anisotropy
state or quality of having different properties along different axes

EXAMPLE An anisotropic transparent particle can show different refractive indices with the vibration direction of incident light.

2.8
asbestiform
specific type of mineral fibrosity in which the fibres and fibrils possess high tensile strength and flexibility

[ISO 13794:1999,[4] 2.6]

2.9
asbestos
term applied to a group of silicate minerals belonging to the serpentine and amphibole groups which have crystallized in the asbestiform habit, causing them to be easily separated into long, thin, flexible, strong fibres when crushed or processed

NOTE 1 The Chemical Abstracts Service Registry Numbers of the *most common* asbestos varieties are: chrysotile (12001-29-5), crocidolite (12001-28-4), grunerite asbestos (amosite) (12172-73-5), anthophyllite asbestos (77536-67-5), tremolite asbestos (77536-68-6) and actinolite asbestos (77536-66-4).

[ISO 13794:1999,[4] 2.7]

NOTE 2 Other varieties of asbestiform amphibole, such as richterite asbestos and winchite asbestos (Reference [20]), are also found in some products such as vermiculite and talc.

2.10
aspect ratio
ratio of length to width of a particle

[ISO 13794:1999,[4] 2.10]

2.11
Bertrand lens
intermediate lens which transfers an image of the back focal plane of the objective into the primary image plane

NOTE The Bertrand lens is used for conoscopic observation in polarized light microscopy and for adjustment of the microscope illuminating system, especially in phase-contrast and modulation-contrast microscopy.

[ISO 10934-1:2002,[3] 2.87.2]

2.12
birefringence
quantitative expression of the maximum difference in refractive index due to double refraction

[ISO 10934-1:2002,[3] 2.16]

2.13
camera length
equivalent projection length between the specimen and its electron diffraction pattern, in the absence of lens action

[ISO 13794:1999,[4] 2.12]

2.14
chrysotile
fibrous mineral of the serpentine group which has the nominal composition:

$Mg_3Si_2O_5(OH)_4$

NOTE Most natural chrysotile deviates little from this nominal composition. In some varieties of chrysotile, minor substitution of silicon by Al^{3+} may occur. Minor substitution of magnesium by Al^{3+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+} and Co^{2+} may also be present. Chrysotile is the most prevalent type of asbestos.

[ISO 13794:1999,[4] 2.13]

2.15
cleavage
breaking of a mineral along one of its crystallographic directions

[ISO 13794:1999,[4] 2.14]

2.16
cleavage fragment
fragment of a crystal that is bounded by cleavage faces

NOTE Crushing of non-asbestiform amphibole generally yields elongated fragments that conform to the definition of a fibre, but rarely have aspect ratios exceeding 30:1.

2.17
crossed polars
state in which the polarization directions of the polars (polarizer and analyser) are mutually perpendicular

[ISO 10934-1:2002,[3] 2.117.2]

2.18
***d*-spacing**
distance between identical adjacent and parallel planes of atoms in a crystal

[ISO 13794:1999,[4] 2.18]

2.19
dispersion
variation of refractive index with wavelength of light

[ISO 7348:1992,[1] 05.03.26]

2.20
dispersion staining
effect produced when a transparent object is immersed in a surrounding medium, the refractive index of which is equal to that of the object at a wavelength in the visible range, but which has a significantly higher optical dispersion than the object

NOTE Only the light refracted at the edges of the object is imaged, and this gives rise to colours at the interface between the object and the surrounding medium. The particular colour is a measure of the wavelength at which the refractive index of the object and that of the medium are equal.

2.21
electron diffraction
technique in electron microscopy by which the crystal structure of a specimen is examined

[ISO 13794:1999,[4] 2.19]

2.22
electron scattering power
extent to which a thin layer of substance scatters impinging electrons from their original directions

[ISO 13794:1999,[4] 2.20]

2.23
energy dispersive X-ray analysis
EDXA
measurement of the energies and intensities of X-rays by use of a solid-state detector and multichannel analyser system

[ISO 13794:1999,[4] 2.22]

2.24
eucentric
condition in which the area of interest of an object is placed on a tilting axis, at the intersection of the electron beam with that axis, and is in the plane of focus

[ISO 13794:1999,[4] 2.23]

2.25
extinction
condition in which an optically anisotropic object appears dark when observed between crossed polars

[ISO 10934-1:2002,[3] 2.51]

NOTE Extinction occurs when the vibration directions of the crystal are parallel to the vibration directions in the polarizer and analyser.

2.26
extinction angle
angle between the extinction position and the position at which the length of a fibre is parallel to the polarizer or analyser vibration directions

2.27
fibril
single fibre of asbestos which cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearances

[ISO 13794:1999,[4] 2.25]

2.28
fibre
elongated particle which has parallel or stepped sides

[ISO 13794:1999,[4] 2.26]

NOTE For the purposes of this part of ISO 22262, a fibre is defined to have an aspect ratio greater than or equal to 3:1.

2.29
fibre bundle
structure composed of parallel, smaller diameter fibres attached along their lengths

NOTE A fibre bundle may exhibit diverging fibres at one or both ends.

[ISO 13794:1999,[4] 2.27]

2.30
gamma refractive index
γ
highest refractive index exhibited by a fibre

2.31
habit
characteristic crystal growth form, or combination of these forms, of a mineral, including characteristic irregularities

[ISO 13794:1999,[4] 2.30]

2.32
high-efficiency particulate air filter
HEPA
filter that is at least 99,97 % efficient by volume on 0,3 µm particles

[ISO 14952-1:2003,[6] 2.13]

2.33
isotropic
having the same properties in all directions

[ISO 14686:2003,[5] 2.23]

2.34
Köhler illumination
method of illuminating specimens in which an image of the illumination source is projected by a collector into the plane of the aperture diaphragm in the front focal plane of the condenser, which then projects an image of an illuminated field diaphragm at the opening of the collector into the specimen plane

2.35
lamda zero
λ_0
matching wavelength corresponding to the dispersion staining colour shown by a particle in an immersion medium

NOTE At this wavelength, the particle and the immersion medium have the same refractive index.

2.36
matrix
material in a laboratory sample within which fibres are dispersed

2.37
Miller index
set of either three or four integer numbers used to specify the orientation of a crystallographic plane in relation to the crystal axes

[ISO 13794:1999,[4] 2.33]

2.38
pleochroism
property of an optically anisotropic medium by which it exhibits different brightness and/or colour for different directions of light propagation, or for different vibrations, on account of variation in selective spectral absorption of transmitted light

2.39
polarized light
light in which the vibrations are partially or completely suppressed in certain directions at any given instant

NOTE The vector of vibration may describe a linear, circular or elliptical shape.

[ISO 10934-1:2002,[3] 2.88.1]

2.40
polarizer
polar placed in the light path before the object

[ISO 10934-1:2002,[3] 2.117.4]

2.41
polar
device which selects plane-polarized light from natural light

[ISO 10934-1:2002,[3] 2.117]

2.42
refractive index
n
ratio of the speed of light (more exactly, the phase velocity) in a vacuum to that in a given medium

[ISO 10934-1:2002,[3] 2.124]

2.43
retardation
difference in optical path length expressed in wavelengths, length units or phase angles between two mutually perpendicular plane-polarized waves

[ISO 10934-1:2002,[3] 2.128]

2.44
selected area electron diffraction
technique in electron microscopy in which the crystal structure of a small area of a sample is examined

[ISO 13794:1999,[4] 2.38]

2.45
serpentine
group of common rock-forming minerals having the nominal formula:

$Mg_3Si_2O_5(OH)_4$

[ISO 13794:1999,[4] 2.39]

2.46
sign of elongation
description of the directions of the high and low refractive indices in a fibre

NOTE The fibre is described as positive when the higher refractive index is parallel to the length of the fibre, and negative when the lower refractive index is parallel to the length of the fibre.

2.47
temperature coefficient of refractive index
measure of the change of refractive index of a substance with temperature

2.48
twinning
occurrence of crystals of the same species joined together at a particular mutual orientation, and such that the relative orientations are related by a definite law

[ISO 13794:1999,[4] 2.41]

2.49
unopened fibre
large diameter asbestos fibre bundle that has not been separated into its constituent fibrils or fibres

[ISO 13794:1999,[4] 2.42]

2.50
zone-axis
line or crystallographic direction through the centre of a crystal which is parallel to the intersection edges of the crystal faces defining the crystal zone

[ISO 13794:1999,[4] 2.43]

3 Symbols and abbreviated terms

$\frac{dn}{dT}$	change of RI of an immersion medium per degree Celsius change of temperature
n_D^{25}	RI of a liquid for the sodium D line (589,3 nm) and at a temperature of 25 °C
α	lowest RI of an anisotropic particle
β	intermediate RI of an anisotropic particle
γ	highest RI of an anisotropic particle
λ_0	wavelength at which the RI of a particle is equal to the RI of the liquid in which it is immersed
ED	electron diffraction
EDXA	energy dispersive X-ray analysis
FWHM	full width, half maximum

HEPA	high-efficiency particle absolute
MEC	mixed esters of cellulose
PC	polycarbonate
PCOM	phase contrast optical microscopy
PLM	polarized light microscopy
RI	refractive index
SAED	selected area electron diffraction
SEM	scanning electron microscopy
TEM	transmission electron microscopy

4 Principle

4.1 General

A suitable tool is used, in compliance with the relevant safety regulations, to take a sample from the material to be analysed. The sample is then appropriately packed and labelled for transportation to the laboratory.

A representative sample of the bulk material is initially examined using a stereo-binocular microscope. Typical fibres are removed using tweezers and mounted in appropriate liquid immersion media on slides for examination by polarized light microscopy. Asbestos fibres are identified based on morphology, colour, pleochroism, and the α (lowest) and γ (highest) refractive indices qualitatively assessed using the dispersion staining technique. Detection of commercial asbestos (chrysotile, amosite, crocidolite or anthophyllite), either alone or in combination, is assumed to indicate that the asbestos is present at a mass fraction exceeding 0,1 %. Optionally, a visual estimate of the asbestos mass fraction is reported in one of several broad mass fraction ranges. Tremolite, actinolite and richterite/winchite are identified by the same procedure, but since they are usually present as contaminants of mineral products, detection of these minerals does not provide information as to their minimum mass fraction. Optionally, fibres may be identified by SEM or TEM.

4.2 Substance determination

This International Standard specifies a number of reference methods for determination of asbestos in solid materials. This part of ISO 22262 provides a method for qualitative analysis of specific commercial products for the presence of asbestos (chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite and richterite/winchite). Other parts of this International Standard provide methods for the analysis of specific types of commercial products for which the use of PLM on the untreated sample yields unacceptable rates of error, and for the quantification of asbestos in the low mass fraction range below approximately 5 %.

4.3 Type of sample

The method specified in this part of ISO 22262 is applicable to sampling and analysis of commercial products from which individual fibres of asbestos can be manually separated from the matrix material, either by picking fibres from surfaces and newly fractured surfaces, or after chemical treatments, acid extraction or ashing, such that the fibres can be identified by one of the specified identification methods. This part of ISO 22262 is generally applicable to asbestos-containing building materials such as fireproofing, thermal pipe and boiler insulations, asbestos cement, plasters, roofing, and other similar materials. The method is also applicable to the identification of asbestos in a range of other industrial minerals and materials.

4.4 Range

Experience from proficiency testing has shown that the range of this part of ISO 22262, when it is applied to a suitably prepared sample in which the asbestos fibres are sufficiently large to be optically visible using a low-

magnification stereomicroscope, is from less than 0,1 % to 100 %. The lower end of the range can be extended downwards by use of appropriate techniques.

4.5 Limit of detection

The limit of detection of this method is defined as the detection and identification of one fibre or fibre bundle in the amount of sample examined. The limit of detection that can be achieved depends on:

a) the nature of the matrix of the sample;

b) the size of the asbestos fibres and bundles;

c) the use of appropriate sample preparation and matrix reduction procedures;

d) the amount of time expended on examination of the sample;

e) the method of analysis used — PLM, SEM or TEM.

With appropriate matrix reduction procedures that are tailored to the nature of the sample, the limit of detection can be significantly lower than 0,01 %.

4.6 Limitations of PLM in the detection of asbestos

The ability to detect and identify asbestos by PLM is limited by the resolution of the optical microscope and sometimes by the masking effects of other materials that comprise the balance of the sample. Asbestos fibres with widths below approximately 0,2 µm are unlikely to be detected by PLM. However, for all varieties of amphibole asbestos, and most varieties of chrysotile, a large proportion of the mass comprises fibres that exceed this width and, because of this, asbestos can be reliably detected by PLM. Accordingly, provided that the nature of the matrix material on the microscope preparation is such that it does not obscure any asbestos fibres that might be present, a non-detected result by PLM indicates that the mass fraction of asbestos is below the limit of detection.

One commercial source of chrysotile presents problems of detection by PLM. Chrysotile originating from the Coalinga deposit in California, USA, contains no fibrils longer than approximately 30 µm and, if these are well dispersed in a sample matrix, the majority of the chrysotile is below the size that can be reliably detected and identified by PLM. The range of application of Coalinga chrysotile is limited to floor tiles, ceiling tiles, drywall joint compounds, mastics, paints, sealants, adhesives, drilling mud, moulded cement building products, and as filler in some plastics. There is a high probability that this variety of chrysotile may not be detected by PLM, even when present in high mass fractions. The size distribution of Coalinga chrysotile makes it unsuitable for most other applications in which asbestos was used and the possibility that it will be encountered in other types of product can generally be discounted. If, on the basis of PLM examination, Coalinga chrysotile is suspected to be present, it is recommended that the sample be examined by electron microscopy.

Asbestos fibres may not be detected by PLM because they are obscured by the matrix of the sample. The matrix reduction methods specified in this part of ISO 22262 are intended to minimize the possibility of failing to detect asbestos in such samples.

5 Sample collection

5.1 Requirements

5.1.1 Sampling apparatus. Depending on the nature of the material to be sampled, an appropriate tool is required for collection of the sample. If the material is soft, such as thermal insulation or fireproofing, a knife or scalpel may be sufficient. In other situations, a cork borer may be used to sample all of the layers of a layered material. If the material is hard, e.g. asbestos–cement, tools such as pliers, a wire cutter, hammer and chisel or rotating hole saw can be needed.

5.1.2 HEPA vacuum cleaner. A HEPA vacuum cleaner, approved for asbestos, is required for cleaning around the sampling location after collection of the sample to minimize dispersion of asbestos-containing dust or particulate matter.

5.1.3 Materials and supplies for sampling.

5.1.3.1 Wetting agent. A wetting agent may be used to limit the generation of airborne dust during the collection of the sample. Water, or water to which a small amount of surfactant has been added, may be applied to the surface before sampling using a spray bottle or brush.

IMPORTANT If a sample is being collected for the purpose of product identification, use no wetting agent, since this may result in alteration of the sample composition by addition of surfactant, and by dissolution and loss of water-soluble constituents.

5.1.3.2 Filler. After collection of the sample, a minor repair may be necessary to seal the damaged area. Depending on the circumstances, spray paint, touch-up paint or plaster may be used.

5.1.3.3 Sample containers. Appropriate dust-tight containers are required for packaging the sample. Plastic bags with "zip" closures or bottles with screw caps may be used.

5.1.3.4 Labels. A method for labelling samples is required. Self-adhesive paper labels may be used. Alternatively, a waterproof marker may be sufficient for field use.

5.1.3.5 Dust mask. A dust mask with filter approved for respiratory protection against airborne asbestos fibres. Approved filters conform to either the National Institute for Occupational Safety and Health (NIOSH) P100 or the European Standard EN 143[9] P3 specification. Other types of personal protective equipment may be used if warranted by the situation.

5.1.3.6 Light. Either a flashlight or an appropriate light source is required for collection of samples in dark locations.

5.1.3.7 Plastic bags. Labelled plastic bags of appropriate size that can be closed tightly and are required to collect the waste generated during sampling. Bags containing waste should be placed inside another tightly closed plastic bag.

5.1.3.8 Cleaning supplies. Cleaning materials, such as disposable paper towels and a supply of water, are required for cleaning sampling tools to avoid cross-contamination between samples.

5.1.3.9 Location identifiers. The use of some means of identifying the precise location from which each sample is taken is recommended, since it may be necessary to resample the material at a later date to resolve discrepancies if they arise. A location identifier is invaluable if the sample collected is found not to be representative of the overall area, such as if the sample has been taken from a patch in a location that has been repaired. A specific colour of spray paint, or appropriate permanent labels applied to the precise location, may be used.

5.2 Procedure

5.2.1 Safety precautions

Handling asbestos is regulated by many jurisdictions, and regulations often specify a variety of procedures to ensure that individuals performing work and those in close proximity are not exposed to excessive concentrations of airborne asbestos. Exceptions from the regulations are generally permitted for some types of activity that are minimally invasive, such as the removal of material samples for analysis.

IMPORTANT — Care is necessary during sampling of materials that may contain asbestos, and precautions should be taken to avoid creating and inhaling airborne asbestos particles when sampling materials suspected of containing asbestos. If the handling instructions in this clause are followed, it may be

assumed that the level of dust meets the thresholds of safety defined in the regulations. In exceptional cases, more extensive precautions may be necessary to prevent the release of airborne fibres.

Sometimes different materials may have been applied to a surface as several layers. It is recommended that samples of all of the individual layers be collected. If a borer or hole-sawing device is used to penetrate several layers, the device should be operated so that it rotates slowly. This ensures that only coarse turnings are produced. High-speed devices are not recommended, since it is then necessary to take more complex safety precautions such as local suction and filtration to collect the dust generated.

5.2.2 Sample size requirements

5.2.2.1 General

Although only a few milligrams of sample are required for the analytical methods specified, it is necessary to take into account the homogeneity of the material, and to ensure that the sample is of sufficient size to be representative of the material under investigation. If inspection shows that the material is finely divided and homogeneous when examined visually, or if the nature of the material is recognized as such from previous knowledge, a minimum sample size of approximately 1 cm^3 generally provides sufficient material for analysis. However, a minimum volume of 10 cm^3 is recommended for materials such as sprayed fireproofing, and as much as 1 000 cm^3 for materials such as loose-fill vermiculite.

5.2.2.2 Representative sample

A wide range of asbestos-containing materials was used in the past. Experience is very valuable in the selection of the materials to be sampled and sampling can be facilitated by the use of all available prior knowledge about the materials or components from which the sample is being collected. It is essential that the sample collected be representative of the composition of the product with respect to its asbestos content. Although many asbestos-containing materials may seem to be homogeneous when visually examined, they can be quite inhomogeneous in the microscopic size range. This is particularly the case for materials such as texture coats, in which the fragments of aggregate are significantly larger than the other constituents of the material.

In some types of material, particularly those that have been mixed at a building site, rather than a commercial product manufactured and mixed under a formulation and quality control procedure, the asbestos may not be distributed homogeneously within the material. For these types of materials, it is necessary to collect a larger sample to ensure that the sample is representative of the material.

It is recommended that a portion of the sample be archived, because further examination of the sample is often the only way in which potential questions can be resolved.

In addition to the problem of inhomogeneity, the possibility that repairs using materials from different sources may have occurred needs to be considered. For example, during renovation or repairs, some asbestos-free ceiling tiles may have been installed in a suspended ceiling, the balance of which contain asbestos, for no other reason than such ceiling tiles were readily available at the time. During repairs or rebuilding, other materials of the same appearance, but having different compositions, may have been used to repair damage to fireproofing, thermal insulation or bulkheads.

It is important to recognize that the analytical result relates only to the actual sample tested. If the sample collected is not representative, the result will not be representative of the material.

Annex A, which lists the asbestos-containing materials most frequently used, provides guidance for identifying different types of material.

5.2.2.3 Number of samples

The number of samples to be taken is dependent on the nature of the material, whether the material is homogeneous or inhomogeneous, and the size of the area under consideration. In the case of materials known from prior experience to be homogeneous, it may be sufficient to collect one sample, although collection of more than one sample provides additional confidence that the results are representative of the material being sampled. When materials are suspected to be inhomogeneous, it is necessary to collect several samples and

to ensure that each of the samples is of sufficient size. If it is intended to determine the range of asbestos content in an area of material, it is necessary to analyse all of the samples individually. Otherwise, such samples may be combined before analysis in order to ensure that the sample analysed represents the mean asbestos mass fraction of the material.

5.2.2.4 Precautions to avoid cross-contamination between samples

It is most important that precautions be taken to ensure that cross-contamination of samples does not occur. Clean all tools used for collecting samples prior to initial use and again after collection of each sample. Use a new and unused container or plastic bag for each sample, and double-bag each sample.

5.2.2.5 Sampling strategy

Selection of the sampling locations depends on the type of area being sampled and on the nature of the product suspected to contain asbestos.

The selection of the sampling locations shall be made in accordance with any national regulations.

The material being sampled may be known to be homogeneous, e.g. a manufactured packing material or sheet material. Samples should be collected at locations that are as inconspicuous as possible. Locations that exhibit prior superficial damage or locations behind readily detached covers are particularly suitable, provided that there are no reasons to suspect that the material in such locations is not representative.

IMPORTANT — Ensure that the sampling location is not at a position where repair using a different material has previously occurred.

If the material under test has a layered structure, e.g. in the case of multilayer pipe insulations or multilayer floor coverings, include all layers of the material in the collected sample. Include any coverings or adhesive layers, such as coatings or glues. Do not attempt to separate the layers under field conditions; separation of individual layers for analysis is best performed under controlled conditions in the laboratory.

If the product under test is behind a wall cladding or other covering, power sockets or light switch recesses are frequently suitable as locations for collection of material samples. If it is not possible to gain access in this manner, it is necessary to cut the claddings or coverings open in order to enable sample collection. These openings should be made at a location that detracts from the visual appearance as little as possible, e.g. behind baseboards.

5.2.2.6 Taking the samples

Release of airborne asbestos fibres from asbestos-containing materials may occur before or during the sampling. The use of containment measures may be necessary. If the material is such that a significant release of airborne asbestos fibres may occur during collection of the sample, sample carefully and moisten the sampling location with water from a spray bottle, a water-soaked brush or a moist paper towel. A moist paper towel is also useful to clean contaminated surfaces after the sample has been collected.

Water should not be used if samples are being collected in the vicinity of operating electrical equipment.

a) For many types of homogeneous material, it is usually possible to collect small amounts of sample without visibly defacing the material and without incurring any significant release of airborne fibres.

b) If the material appears to be homogeneous, collect a sample area more than 1 cm^2 in the case of thin materials, or a volume greater than 1 cm^3 in the case of materials having a thickness of several centimetres. Remove the sample by breaking it off with pincers or preferably using a sharp cutting tool. If the material appears to be inhomogeneous, collect a sufficient amount of sample to give confidence that the volume of sample is representative of the material.

c) Place each sample in an individual dust-tight container.

d) Wipe the sampling site and the immediate surroundings, keeping them moist, or clean the area around the sample location using a vacuum cleaner with a HEPA filter.

e) If necessary, seal the exposed surface from which the sample was taken using touch-up paint, glue or other appropriate sealant.

f) Affix, if applicable and agreed to by the facility administration, a permanent identification marker to the exact location from which the sample is removed.

5.2.2.7 Sample labelling

Label the sample container clearly, either by using a permanent marker pen or by attaching a permanent adhesive label. Confirm that the sample label corresponds to the information on any identification marker affixed to the sampling location.

5.2.2.8 Sampling record

Make a record of the sample that contains at least the following information:

a) full description of the type of material;

EXAMPLE Thermal insulation, board, floor tile.

b) all details recorded on the sample label;

c) precise description of the sampling location;

d) building identification;

e) identification of the room (if applicable);

f) location in the room from which the sample was collected;

g) the date that the sample was collected;

h) the name of the person who collected the sample;

i) whether the sample is a composite derived from the combination of separately collected samples;

j) whether the sample is a multilayer sample — for multilayer samples, the positions of each of the relevant layers shall be noted.

If the sampling location is not adequately specified by the details specified in a) to f), then, in addition:

k) make a sketch or take a photograph (record the number of the photograph); or, record the position from which the sample was taken on a plan of the building (the drawing identification shall also be noted in the record);

l) report any other relevant data that are available with respect to the sample.

An example of a suitable sampling record is shown in Annex G.

5.2.2.9 Chain of custody

If there is any possibility that the results of sampling and analysis will be subject to litigation or legal scrutiny, it is most important that records be made of all transfers of samples between individuals, starting with the individual who collected the samples through to acceptance of the samples by the analyst. A chain of custody form shall be used for this purpose, on which the date of each transfer and the name of each individual who has relinquished or accepted possession of the samples are recorded.

5.2.2.10 Storage and transport

The samples shall be packaged in dust-tight containers (double if necessary) and a label shall be affixed to the package of samples, indicating that they may contain asbestos. Take care to ensure that unauthorized persons do not have access to the samples. There are no special requirements with respect to climate conditions

for storage and transport of the samples. After the samples have been analysed, they shall be archived for whatever period of time is specified by the individual submitting them to the analytical laboratory.

6 Sample preparation

6.1 General

It is sometimes not possible to identify asbestos in bulk materials because of interference by other constituents, either because the mass fraction of asbestos is too low or because the asbestos is so inhomogeneously distributed that a large amount of the sample would need to be examined in order to reliably detect the asbestos that is present. In these cases, various chemical or physical preparation methods can be used prior to the microscopic examination to remove a large proportion of the non-asbestos constituents, thus facilitating the detection of asbestos in the smaller amount of material that remains.

6.2 Removal of organic materials by ashing

Chrysotile is often difficult to detect when mixed with large amounts of cellulose, or if it is well dispersed in organic matrices such as asphalt or poly(vinyl chloride) (PVC). Also, some other organic fibres such as spider webs and wool have optical properties similar to those of chrysotile. Ashing of the sample at a temperature of 485 °C for a period of approximately 10 h removes the organic constituents with very little effect on the optical properties of chrysotile. Although the colour and optical properties of amosite and crocidolite are altered by this oxidation treatment as a consequence of conversion of some ferrous iron [Fe(II)] to ferric iron [Fe(III)], many of the fibres can often still be identified by PLM. The optical properties of tremolite, actinolite, anthophyllite and richterite/winchite are almost unaffected by this treatment. The heat treatment does not otherwise affect the composition of any of the asbestos varieties, and they can all be identified by electron microscopy after the treatment.

6.3 Removal of soluble constituents by acid treatment

Matrix constituents such as calcite and gypsum often coat asbestos fibres so that their optical properties cannot be reliably examined. These constituents also often constitute a large proportion of the sample mass. Stirring of a sample in 2 mol/l hydrochloric acid for approximately 15 min removes many matrix constituents, and this improves the ability to identify and quantify asbestos. The acid treatment slightly reduces the refractive indices of chrysotile, and it is necessary to account for this when identifying chrysotile by PLM. Do not heat chrysotile in acid at temperatures exceeding 60 °C. This acid treatment does not affect the optical properties of any of the other asbestos varieties.

6.4 Sedimentation and flotation

Some materials contain large sizes of aggregate or sand that can be separated in water suspension by sedimentation or flotation. A large proportion of constituents such as vermiculite or perlite can be separated by flotation. Sand or small solid aggregate sediment in water much more rapidly than most of the asbestos, and in some samples a large proportion of the sand or aggregate can be separated from the fraction that contains any asbestos.

6.5 Combination of gravimetric reduction procedures

The procedures specified in 6.2, 6.3 and 6.4 may be combined as appropriate for the particular sample.

It is generally recommended that the procedures be used sequentially in the order given.

7 Analysis by PLM

7.1 Requirements

7.1.1 Stereo-binocular microscope, for initial observation of samples. The examination is facilitated if the microscope has a continuous range of magnification from approximately 10× to 40×.

7.1.2 Polarized light microscope, capable of Köhler (or Köhler-type) illumination is needed for fibre identification. The following optical accessories are necessary:

a) light source with blue “daylight” filter;

b) focusing sub-stage condenser with a numerical aperture (NA) greater than or equal to that of the objective in use, with a field-limiting adjustable aperture;

c) focusing ocular with magnification of 10 times or 12 times, with a cross-hair graticule;

d) strain-free objectives with magnifications of 4 times, 10 times, and 40 times or similar magnifications;

e) polarizer and removable analyser, the vibration directions of which can be adjusted such that they are at 90° to each other, and can be aligned with the cross-hair in the focusing ocular;

f) slot between the polarizer and analyser to allow accessory plates to be inserted at an angle of 45° to the polarizer and analyser vibration directions;

g) removable retardation plate with approximately 530 nm retardation, with known slow and fast vibration directions;

h) dispersion staining objective with magnification of 10 times or 40 times, or a demonstrated functional equivalent (MDHS 77[11]);

i) Bertrand lens or a focusing telescopic ocular to allow observation of the back focal plane of the objective lens;

j) level rotating specimen stage for which the centre of rotation can be centred relative to the optical axis of the microscope for each of the objective lenses.

7.1.3 Dust extract hood. Handling and manipulation of bulk materials suspected to contain asbestos shall be performed in a suitable dust extract hood, so that neither the analyst nor the laboratory environment is exposed to airborne asbestos fibres.

7.1.4 Sample preparation.

7.1.4.1 Refractive index liquids. The majority of commercial asbestos-containing products contain only chrysotile, amosite or crocidolite, or mixtures of these three types of asbestos. Identification of these three types of asbestos can be achieved using liquids of RI 1,550, 1,680 and 1,700. The RI values of these liquids are specified for light of wavelength 589,3 nm (sodium D line) at a temperature of 25 °C.

For identification of tremolite, actinolite, anthophyllite and richterite/winchite, RI liquids in the range 1,605 to 1,660 are required, at intervals of 0,005.

Suitable calibrated RI liquids are commercially available, and a set of liquids with RIs from 1,500 to 1,700, at intervals of 0,005, gives sufficient range and discrimination.

If commercially available RI liquids cannot be obtained, a set of liquids sufficient for use in this part of ISO 22262 can be prepared (References [16][21]) using common chemical reagents as specified in Table 1.

Table 1 — Reagents for preparation of RI immersion media

Reagent	n_D^{25}	$\frac{dn}{dT}$
Glycerol triacetate	1,427 7	–0,000 48
Ethyl cinnamate	1,557 4	–0,000 48
Bromobenzene	1,557 0	–0,000 54
Iodobenzene	1,617 3	–0,000 54
1-Chloronaphthalene	1,630 4	–0,000 44
1-Bromonaphthalene	1,658 0	–0,000 45
1-Iodonaphthalene	1,700 4	–0,000 44
Diiodomethane	1,739 0	–0,000 70
Commercially available RI media, and the reagents listed here, should be used in accordance with applicable safety precautions.		

Table 2 shows the mixtures of reagents required to prepare a set of RI immersion media. The three primary RI liquids for identification of chrysotile, amosite and crocidolite are indicated in Table 2 in bold type (1,550, 1,680 and 1,700). Tremolite, actinolite or anthophyllite can often be identified using only RI liquids 1,605 and 1,630, also indicated in Table 2 in bold type. Tremolite, actinolite or anthophyllite may be encountered in which the refractive indices are high because of increased iron mass fraction, and use of other RI liquids in Table 2 may be necessary in order to assess the refractive indices.

Table 2 — Mixtures and single compounds required for RI liquids

Liquid n_D^{25}	Liquid 1	Volume fraction, liquid 1 %	Liquid 2	Volume fraction, liquid 2 %	$\frac{dn}{dT}$
1,545	Ethyl cinnamate	90,44	Glycerol triacetate	9,56	–0,000 48
1,550	**Ethyl cinnamate**	**94,30**	**Glycerol triacetate**	**5,70**	**–0,000 48**
1,555	Ethyl cinnamate	98,15	Glycerol triacetate	1,85	–0,000 48
1,560	Bromobenzene	95,03	Iodobenzene	4,97	–0,000 54
1,605	**Iodobenzene**	**79,60**	**Bromobenzene**	**20,40**	**–0,000 54**
1,610	Iodobenzene	87,89	Bromobenzene	12,11	–0,000 54
1,615	Iodobenzene	96,19	Bromobenzene	3,81	–0,000 54
1,620	1-Chloronaphthalene	85,83	Bromobenzene	14,17	–0,000 45
1,625	1-Chloronaphthalene	92,64	Bromobenzene	7,36	–0,000 45
1,630	**1-Chloronaphthalene**	**100**	—	—	**–0,000 44**
1,635	1-Bromonaphthalene	78,99	Bromobenzene	21,01	–0,000 47
1,640	1-Bromonaphthalene	84,05	Bromobenzene	15,95	–0,000 46
1,645	1-Bromonaphthalene	89,11	Bromobenzene	10,89	–0,000 46
1,650	1-Bromonaphthalene	94,18	Bromobenzene	5,82	–0,000 46
1,655	1-Bromonaphthalene	99,24	Bromobenzene	0,76	–0,000 45
1,660	1-Bromonaphthalene	90,48	1-Iodonaphthalene	9,52	–0,000 45
1,680	**1-Iodonaphthalene**	**54,31**	**1-Bromonaphthalene**	**45,69**	**–0,000 44**
1,700	**1-Iodonaphthalene**	**100**	—	—	**–0,000 44**

7.1.4.2 Asbestos reference standards. Asbestos reference standards are required. Suitable sets of standards are SRM 1866[1)] (chrysotile, crocidolite and amosite) and SRM 1867[1)] (tremolite, actinolite and anthophyllite) from the US National Institute of Standards and Technology (NIST), see Table 3, or from the UK Health and Safety Executive (HSE) [Chrysotile (Canada and Zimbabwe), crocidolite, amosite, tremolite, actinolite and anthophyllite][2)] see Table 4. SRM 1867 tremolite and actinolite are particularly useful for qualitative discrimination between tremolite and actinolite. The International Mineralogical Association (IMA) (References [23][24]) has specified that values of the mass fraction ratio Mg/(Mg + Fe) below 0,9 are defined as tremolite, and those above 0,9 are defined as actinolite. SRM 1867 tremolite has a value of 0,84, and SRM 1867 actinolite has a value of 0,94, providing reference samples representing compositions just below and just above the IMA boundary. It is important to recognize that the IMA boundary between tremolite and actinolite is only a convention within a continuum of composition in which the iron and magnesium mass fractions vary in a reciprocal manner.

Table 3 — Optical properties of SRM 1866 and SRM 1867 reference asbestos samples

Property	Chrysotile	Amosite	Crocidolite	Anthophyllite	Tremolite	Actinolite
Colour	White	Grey–brown	Blue	Light brown	White	White
Pleochroism	None	Very weak	α: Blue, γ: grey	None	None	None
Birefringence	Low	Medium	Low	Medium	Medium	Medium
Sign of elongation	Positive	Positive	Negative	Positive	Positive	Positive
Extinction	Parallel	Parallel	Parallel	Parallel	16,6°	15,9°
γ	1,556	1,701	—[a]	1,636	1,634	1,639
α	1,549	1,679	—[a]	1,615	1,606	1,613

[a] For crocidolite, the certificate of analysis states: "Because strong absorption in the visible light range results in anomalous dispersion characteristics that would not be useful to the analyst, no certified values of refractive index are reported for riebeckite".

Table 4 — Optical properties of HSE reference asbestos samples

Property	Chrysotile (Canada)	Chrysotile (Zimbabwe)	Amosite	Crocidolite	Anthophyllite	Tremolite	Actinolite
Colour	White	White	Grey–brown	Blue	White	White	Pale green
Pleochroism	None	None	Very weak	α: Blue, γ: grey	None	None	γ-Green, α: grey
Birefringence	Low	Low	Medium	Low	Medium	Medium	Medium
Sign of elongation	Positive	Positive	Positive	Negative	Positive	Positive	Positive
Extinction	Parallel	Parallel	Parallel	Parallel	Parallel	Parallel	Parallel
γ	1,552	1,552	1,692	1,696	1,624	1,632	1,652
α	1,544	1,544	1,676	1,688	1,608	1,616	1,644

NOTE The data for the HSE reference asbestos samples notes that, "as with all natural minerals, the reference samples may contain traces of other minerals. In particular, the anthophyllite asbestos sample contains a fibrous variety of talc which may be distinguished by its ribbon-like morphology and generally lower refractive indices".

For those laboratories that are unable to obtain either the NIST or the HSE reference asbestos samples, the Union Internationale Contre le Cancer (UICC) standard reference samples of asbestos (Reference [25]) may be used, see Table 5. These samples were widely distributed internationally, and can still be obtained.

1) Example of a suitable product available commercially from the US National Institute of Standards and Technology (NIST). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

2) Example of a suitable product available commercially from the from the UK Health and Safety Executive (HSE). See Reference [22]. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

However, since the UICC samples were prepared for use in animal studies, they were milled to very small fibre sizes. Also, the UICC samples do not include either tremolite or actinolite.

Table 5 — Optical properties of UICC reference asbestos samples

Property	Chrysotile (Canada)	Chrysotile (Zimbabwe)	Amosite	Crocidolite	Anthophyllite
Colour	White	White	Grey–brown	Blue	White
Pleochroism	None	None	Very weak	α: Blue, γ: grey	None
Birefringence	Low	Low	Medium	Low	Medium
Sign of elongation	Positive	Positive	Positive	Negative	Positive
Extinction	Parallel	Parallel	Parallel	Parallel	Parallel
γ	1,545–1,560	1,553	1,701	1,702	1,620
α	1,545–1,557	1,546	1,679	1,694	1,605

NOTE 1 A range of refractive indices is quoted for the UICC Canadian chrysotile sample. This sample was prepared by blending chrysotile from a number of different mines. Fibres with refractive indices within the approximate ranges specified are present, with birefringence ($\gamma - \alpha$) approximately 0,01.

NOTE 2 The anthophyllite sample also contains a fibrous variety of talc.

7.1.4.3 Sample comminution equipment. An agate mortar and pestle is required for grinding samples to suitable sizes for PLM examination.

7.1.4.4 Microscope slides, 75 mm × 25 mm.

7.1.4.5 Microscope cover glasses, 22 mm × 22 mm. Match the thickness of the cover glasses with that specified by the objective lenses. A thickness of 0,17 mm is required by many commercial objectives.

7.1.4.6 Thermometer, required to measure the temperature of the microscope slide preparation during observation if accurate refractive indices of asbestos fibres are to be recorded.

7.1.4.7 Alcohol or gas burner. A laboratory burner is sometimes useful for discriminating between organic fibres and asbestos fibres.

7.1.4.8 General laboratory supplies. The following supplies and equipment, or equivalent, are required:

a) glassine paper sheets, approximately 15 cm × 15 cm, for examination of samples;

b) scalpel holder and replacement disposable scalpel blades;

c) sampling utensils, including tweezers, needles and spatulas;

d) distilled water;

e) concentrated hydrochloric acid, reagent grade;

f) crucibles, silica or glazed porcelain, with lids;

g) Petri dishes;

h) disposable pipettes;

i) glass filtration assembly, 25 mm or 47 mm diameter;

j) polycarbonate filters, 0,4 µm pore size, 25 mm or 47 mm diameter.

7.1.4.9 Muffle furnace (optional). For ashing of samples to remove interfering organic constituents, a muffle furnace with a temperature range up to 500 °C and a temperature stability of ±10 °C is recommended.

7.1.4.10 Magnetic stirrer (optional). For removal of acid-soluble interfering constituents, a magnetic stirrer with a glass or plastic-coated magnetic stir bar.

7.2 Qualitative analysis by PLM

7.2.1 Calibration

It is essential that the optical components of the PLM be fully understood by the analyst and that the analyst be familiar with the alignment procedure. The alignment of the PLM shall be confirmed prior to conducting any analyses. The designs of microscopes vary and the alignment instructions provided by the manufacturer should be followed. The critical aspects of the alignment are listed in a) to e).

a) The illumination source and sub-stage condenser shall be adjusted so that the field-limiting aperture is in focus (Köhler or Köhler-like illumination).

b) The centre of rotation of the specimen stage shall be aligned with the optical axis of the PLM for each of the objective lenses. This is necessary so that a particle at the centre of the field of view remains at the centre of the field of view during rotation of the stage. This condition is often achieved by centring the rotation for one objective lens, and then laterally adjusting the position of each of the other objective lenses to align their axes with the centre of the stage rotation.

c) The vibration directions of the polarizer and analyser shall be at 90° to each other.

d) The vibration directions of the polarizer and analyser shall accurately coincide with the directions of the cross-hair in the ocular. This can be accomplished using a well-formed birefringent crystal with a known zero extinction angle. Alternatively, orientation plates consisting of an accurately mounted crystal with a fiducial line are commercially available. If the microscope has eyepieces that can be freely rotated, fix the position of the eyepiece containing the cross-hair using adhesive tape, for example.

e) If a mechanical stage is installed on the rotating stage, the directions of the mechanical stage should be adjusted such that the zero angular position of the rotation stage corresponds to lateral motions of the mechanical stage parallel to the polarizer and analyser directions.

On the initial set-up of the PLM, the vibration direction of the polarizer and the orientation of the vibration directions of the 530 nm retardation plate shall be determined. The vibration direction of the polarizer can be determined by examination of a slide preparation of crocidolite with the polarizer in position and the analyser withdrawn. Under these conditions, the direction of the length of the crocidolite fibres when the dark blue pleochroism is displayed is the vibration direction of the polarizer. The orientation of the vibration directions of the 530 nm retardation plate can be determined by examination of a fibre of a known reference material such as amosite or chrysotile, and observing the change of interference colour when the retardation plate is inserted. The slow vibration direction of chrysotile or amosite is parallel to the length of the fibre. If the retardation plate adds to the retardation caused by the fibre, the slow vibration directions of the fibre and the retardation plate are parallel. An interference colour chart is provided in Annex B.

Before using RI liquids for the identification of asbestos, even if certified liquids are purchased, it is recommended that the refractive indices of liquids be confirmed using reference glass samples or a refractometer. If kept tightly capped, the refractive indices of these liquids remain stable for at least 2 years. Some RI liquids degrade when exposed to light, therefore they should be stored in dark bottles, preferably in a dark place.

7.2.2 Sample preparation

For many samples, including fireproofing, thermal insulation and asbestos cement products, fibres that can be removed with tweezers are visible during stereomicroscope examination. Mount typical suspected asbestos fibres on a microscope slide and add a drop of the RI liquid appropriate for the suspected asbestos variety. If the suspected asbestos variety cannot be confirmed using the appropriate RI liquid, mount additional fibres from the sample on slides using RI liquids appropriate for the other asbestos varieties.

7.2.3 Sample analysis

7.2.3.1 Analytical sequence

The analytical techniques described have been shown to give reliable and reproducible results. Alternative methods can be used if their equivalence in terms of detection and identification can be demonstrated. Identification of the asbestos fibres should be based on the following analytical sequence:

a) make a preliminary visual examination of the whole of the laboratory sample to assess the sample type and the required sample treatment (if any) — where possible, take a representative test portion at this stage for direct examination by PLM;

b) carry out any required sample treatment to release or isolate fibres;

c) perform a detailed and thorough search under the stereomicroscope to classify the suspected fibre types present;

d) mount representative fibres in appropriate RI liquids on microscope slides;

e) identify the different fibrous components using PLM.

If no asbestos is detected by these procedures, prepare additional slides using random test portions of a few milligrams and search for thin asbestos fibres using PLM.

7.2.3.2 Preliminary examination

Examine the entire sample visually to describe the type of material or product present, and to establish whether there are visible fibres. Note the nature of any matrix materials, as this may indicate the type of treatment required for the sample. Examine the sample using the stereomicroscope. So far as possible, make an initial determination of the number of fibre types present. Record the appearance, colour and texture of the sample and any fibre types observed. For inhomogeneous or layered samples, it may be necessary to describe each separate layer or part of the sample. Sample preparation and the analysis of the sample are dependent on the quality of the initial visual examination. Also, adequate description of the appearance of the sample is important in establishing whether asbestos is present, or in which part of the sample asbestos is present.

7.2.3.3 Sample treatment

The purpose of any initial treatment of laboratory samples is to release fibres from any matrix and to remove fine particles adhering to the fibres (both of which obscure the optical effects and hinder the identification). It is necessary to break non-friable samples (with tools if necessary) and then to examine newly fractured edges using the stereomicroscope to observe any protruding fibres. If samples contain large pieces of hard materials, grinding the sample may be necessary. Surfaces and edges of hard materials may be abraded to release fibres for examination. Routine procedures used for sample treatment should be fully documented. Any deviations from these procedures for particular samples should be recorded.

Dilute acetic acid or cold dilute hydrochloric acid may be used to remove calcium carbonate (limestone), calcium sulfate (gypsum), and calcium silicate, which are commonly used as binders (e.g. for insulation and asbestos boards) and fillers (e.g. in floor tiles). The removal of calcium magnesium carbonate (dolomite) requires the use of cold concentrated hydrochloric acid. Sufficient acid should be added in small aliquots for several minutes or until effervescence stops. Fibre release may be aided by stirring or by ultrasonic treatment. The sample is then filtered and repeatedly washed with water. Residual acid may degrade the fibres and affect the optical properties, and small crystals of salts may form. The sample may be rinsed with ethanol or other volatile solvents to reduce the drying time.

Organic matrices such as plastics, asphalt, resins or rubber products may require prolonged treatment in solvents to remove the matrix. An effective solvent for any particular sample type can be established only by individual testing or by foreknowledge of the type of matrix. Organic matrices may be removed by treatment in a muffle furnace at 485 °C. However, heating may modify the optical properties of some of the asbestos fibres.

7.2.3.4 Stereomicroscope examination

The original samples or portions of sample that have undergone sample treatment should be examined using the stereomicroscope. For many asbestos-containing materials, asbestos fibres can be detected at magnifications within the range of the stereomicroscope. For other types of asbestos-containing material, it may not be possible to detect asbestos fibres using the stereomicroscope. The aim is to detect small fibre bundles, or individual fibres, and tentatively to assign fibre types based on their appearance. This is usually achieved by placing the sample on a piece of glassine paper or in a suitable container and carrying out a detailed search of the entire sample using needles or tweezers to separate the different fibrous components from the matrix. The appearance of these fibres is then noted. The care and vigilance with which the sample is examined at this stage are important in detecting trace quantities of asbestos. Representative fibres or fibre bundles are then selected and mounted for PLM examination.

Describe layered samples by their appearance, and note each distinct layer as a separate entity. Regulations in some jurisdications require that distinct layers be analysed and reported separately. Other types of inhomogeneous sample will require detailed visual examination of all the different phases observed.

Asbestos is generally recognized by the fineness of its fibres, which are most often present as closely packed bundles of fibrils that will divide along their length when pressure is exerted on them with a probe or tweezers. An analyst will rapidly become familiar with characteristics such as distinctive surface lustre, flexibility, and tensile strength. Initial tentative identification of suspected asbestos fibres at this stage will be confirmed or refuted by subsequent examination using PLM, SEM or TEM.

7.2.3.5 Preparation of samples for PLM examination

A tentative identification based on the stereomicroscope evaluation is used to select the most appropriate RI mounting liquid. Fibres selected shall be dry and relatively free from other particulate matter. Representative fibres or fibre bundles are chosen and are placed on a clean microscope slide into a drop of RI liquid, and a clean cover glass is lowered gently onto the slide, avoiding trapping of air bubbles. The RI of the liquid selected should be 1,550 for suspected chrysotile, 1,680 for suspected amosite, 1,700 for suspected crocidolite, 1,605 for suspected tremolite or anthophyllite, and 1,630 for suspected actinolite or richterite/winchite.

If no fibres have been seen in the bulk sample using the stereomicroscope, or no asbestos fibres have been identified by PLM, then tweezers or probes should be used to take random test portions, after the laboratory sample has undergone suitable treatment (if necessary). At least two microscope slide preparations should be made with appropriate RI liquids for examination by PLM. Any large agglomerates should be teased apart with tweezers or needles, or sheared gently between two microscope slides, to give an even distribution of particles. Selection of large particles or fibre bundles may cause tilting of the cover slip and should be avoided. The amount of sample distributed should be such that the appearance and properties of individual fibres are not obscured by other particles.

7.2.3.6 Identification of asbestos by PLM and dispersion staining

Identification of a single asbestos fibre requires the observation of the following properties in the stated observation modes:

a) morphology — observed in all illumination conditions;

b) colour and pleochroism — observed in plane polarized light;

c) birefringence — observed with crossed polars;

d) extinction characteristics — observed with crossed polars;

NOTE The extinction characteristics can also be observed with crossed polars and a 530 nm retardation plate inserted. Under these conditions, when the interference colour of the fibre matches the background colour, the fibre is at the extinction position.

e) sign of elongation — observed with crossed polars and a 530 nm retardation plate inserted;

f) refractive indices — assessed using a dispersion staining objective with polarizer only inserted.

The above order of observations facilitates the assessment of the morphological and optical properties in a logical sequence. Adjust the microscope to give Köhler illumination, centre the stage, and insert the polarizer (usually adjusted to the east–west orientation below the condenser. Under these conditions, observe the morphology and colour of the selected fibre. Rotate the stage and observe whether the fibres are pleochroic. Insert the analyser to give crossed polars, and rotate the stage to observe birefringence and whether the extinction angle is parallel to the length of the fibre or oblique. With the polars still crossed, insert the 530 nm retardation plate and rotate the stage to determine the sign of elongation. Finally, examine the fibre under dispersion staining conditions to assess the refractive indices for parallel and normal vibration directions. This may be achieved by observing the dispersion colours at the interface between the fibre and the RI liquid. Withdraw the analyser and the 530 nm retardation plate, increase the illumination, and insert a dispersion staining objective with a central stop in the back focal plane. Adjust the condenser aperture until the field of view becomes dark. View the back focal plane of the objective using either a Bertrand lens or a telescope ocular, and adjust the condenser alignment until the central beam is obscured by the central stop of the lens.

For fibres that exhibit parallel extinction, record the dispersion staining colours with the fibre parallel to the polarizer vibration direction and normal to the polarizer direction. If fibres exhibit oblique extinction, it is necessary to search for fibres that exhibit the maximum extinction angle. This can be achieved either by scanning the slide for such a fibre or by rotating fibres about their axes by touching the top of the cover slip with a needle. It is only in this orientation that a monoclinic fibre exhibits the γ and the α refractive indices. When such a fibre has been located, record the dispersion staining colours with the fibre at both extinction positions.

In practice, any other sequence may be used provided that all of the required properties are observed. For example, if it is difficult to locate any suspected asbestos fibres on the prepared mount because the sample is dominated by non-asbestos fibres, or if a random sample is being searched, the sample should be scanned with the microscope in the crossed polars condition to detect the asbestos fibres. The sign of elongation may also be observed by interpretation of the observed dispersion staining colours.

The observations made of the morphology and the optical properties of the fibre are recorded. Identification is based on comparing the recorded observations on the fibres selected for analysis (and mounted in the appropriate RI liquid) against the properties of asbestos reference standards. The compositions and optical properties of commercial chrysotile, amosite and crocidolite do not vary significantly, and therefore a close match between the optical properties of the sample fibre and the asbestos standard is normally achieved. Further representative fibres will need to be examined if the observations are inconclusive, or if more than one type of fibre was found in the stereomicroscopic or PLM analysis. For tremolite, actinolite and anthophyllite, the iron mass fraction can vary significantly from one source to another; higher iron mass fractions result in higher refractive indices. Examples of this variability can be seen by comparing the tremolite, actinolite and anthophyllite samples from the SRM 1867 and HSE sets of reference standards, as illustrated in Annex D.

7.2.3.7 Identification of asbestos

7.2.3.7.1 Morphology

A detailed description for the morphology that is characteristic of asbestos is as follows. This morphology is characteristic of the larger fibres seen in stereomicroscope examinations and of fibres selected from laboratory samples for PLM identification of fibre type.

In the light microscope, the asbestiform habit is generally recognized by the following characteristics:

a) the presence of fibre aspect ratios in the range of 20:1 or higher for fibres longer than 5 µm;

b) the capability of longitudinal splitting into very thin fibrils, generally less than 0,5 µm in width;

c) in addition, observation of any of the following characteristics for the fibre type under consideration provides additional confirmation that the fibres are asbestiform:

   1) parallel fibres occurring in bundles,

   2) fibre bundles displaying splayed ends,

   3) fibres in the form of thin needles,

4) matted masses of individual fibres,

5) fibres showing curvature.

In practice, if chrysotile, crocidolite or amosite is identified in a commercial product, the assumption can safely be made that the fibres are asbestiform and that these fibres conform to the description above. This assumption can be made because these three types of asbestos were mined and processed to yield fibres with specific properties for intentional incorporation into products. Some anthophyllite asbestos was used in a few commercial products, but very little was mined and used commercially. Tremolite asbestos has been found in some surfacing and fireproofing applications in Japan. However, other than these occurrences, the amphiboles tremolite, actinolite, and richterite/winchite were not generally used in commerce, and their presence in a product is more likely a consequence of naturally occurring contamination of one or more of the major constituents. Accordingly, no assumption can be made as to whether the amphibole is asbestiform or non-asbestiform. Anthophyllite can occur as contamination of other mineral products, and in such situations no assumption can be made as to whether it is asbestiform or non-asbestiform. In some samples, these amphiboles may exhibit a mixture of morphological types, and quantitative determination of the regulatory status of such samples may require a detailed study of the fibre size distribution that is beyond the scope of this part of ISO 22262.

In general, for this part of ISO 22262, the presence of either the asbestiform or the non-asbestiform analogues of tremolite, actinolite, anthophyllite or richterite/winchite can usually be specified. If the majority of the amphibole fibres longer than 5 µm have aspect ratios equal to or lower than 5:1, and if the fibres do not exhibit any of the characteristics in c), it can be concluded that the amphibole is probably non-asbestiform, with the degree of certainty increasing with decreasing maximum aspect ratio. If any amphibole fibres longer than 5 µm with aspect ratios in the range of 20:1 or higher are observed, then it can be concluded that amphibole asbestos is probably present, with the degree of certainty increasing with increasing aspect ratio.

NOTE This is intended as guidance for analysts to discriminate between non-asbestiform and asbestiform amphibole populations. It is not intended to override the definition of asbestos as presented in 2.9 nor to override any national regulation.

It is necessary to appreciate that some samples may still present ambiguities with respect to discrimination between asbestiform and non-asbestiform analogues, and such ambiguities, when observed, shall be reported as part of the results.

7.2.3.7.2 Colour and pleochroism

Colour and pleochroism are observed using plane polarized light. Pleochroism is a diagnostic property in the identification of crocidolite. Crocidolite has a strong absorption, which gives a dark blue colour when the fibres are parallel to the polarizer vibration direction, changing to pale blue or grey when the fibres are perpendicular to the polarizer vibration direction. This is illustrated in Figures D.13 and D.14. Pleochroism in amosite may occur after heating, or occasionally in unheated fibres, depending on the Fe/Mg mass fraction ratio of the mineral. Chrysotile shows little colour contrast and no pleochroism in plane polarized light. Depending on the iron mass fraction, actinolite may exhibit a green colour when the fibres are parallel to the polarizer vibration direction, changing to a grey or yellowish colour when the fibres are perpendicular to the polarizer direction. Pleochroism in the HSE actinolite reference sample is illustrated in Figures D.43 and D.44.

7.2.3.7.3 Birefringence

When a particle with more than one RI is observed between crossed polars with its planes of vibration at 45° to those of the polarizer, interference colours are observed against the dark background. For asbestos, these interference colours depend on the fibre thickness, the birefringence and on the degree of randomness of the fibril orientation about the fibre axis.

Between crossed polars, an asbestos fibre aligned at 45° to the polarizer vibration direction should be clearly visible. Chrysotile has a low birefringence and gives a grey colour for thin fibres, and a white colour or higher first (or even second) order colours for thick fibres. Crocidolite has a low birefringence and anomalous interference colours caused by strong absorption in the visible light range. Amosite has moderate birefringence, giving white interference colours for thin fibres and higher first or second order colours for thick fibres. Tremolite, actinolite and anthophyllite, and richterite/winchite similarly exhibit moderate birefringence. Fibres with a variable thickness, e.g. with wedge-shaped cross-sections, show parallel bands of colour along their lengths, representing lower interference colours for progressively thinner sections. Examples are shown in Annex D.

Isotropic materials have zero birefringence, and therefore do not exhibit interference colours. Between crossed polars, isotropic materials such as man-made vitreous fibres are almost invisible, but, depending on the difference between their RI and that of the immersion liquid, are often seen easily with the 530 nm retardation plate in position or with slightly uncrossed polars. Interference colours can be used to distinguish asbestos from some natural organic fibres, which may show non-uniform interference along the fibre length and also incomplete extinction.

7.2.3.7.4 Extinction angle

As the microscope stage is rotated through 360°, an asbestos fibre viewed between crossed polars disappears from view or "extinguishes" at four positions, each 90° apart, while at an angle of 45° to an extinction position, interference colours should be visible. Many fibres, including asbestos, generally show complete extinction when parallel to the vibration directions of the polarizer or the analyser. Chrysotile, amosite, crocidolite and anthophyllite each show parallel extinction when the fibre is parallel to the vibration direction of the polarizer or analyser. Tremolite, actinolite, and richterite/winchite may exhibit parallel extinction or oblique extinction, depending on the orientation of the fibre and the crystalline nature of the fibre. Highly asbestiform fibres of these amphiboles may show parallel extinction at all axial orientations. Other fibres of high aspect ratio may show oblique extinction, and axial rotation of the fibre by touching the cover glass of the slide with a needle allows the maximum extinction angle to be determined. Tremolite and some low-iron actinolite fibres that exhibit only parallel extinction cannot easily be discriminated from anthophyllite. However, it is unlikely that *all* of the tremolite or actinolite fibres in a sample would exhibit parallel extinction, and observation of some with oblique extinction angles would confirm the identity of the mineral, with the presumption that parallel extinction fibres with otherwise similar properties are the same mineral species. In these cases, reliable discrimination between anthophyllite and either tremolite or actinolite may only be possible by examination of the compositions of the fibres by SEM or TEM.

7.2.3.7.5 Sign of elongation

The sign of elongation describes the relationship between the length of the fibre and the optical properties. For asbestos fibres the two available vibration directions are parallel to the long axis and perpendicular to it. If the high RI vibration direction is parallel to the long axis, then the fibre is described as positive; if the low RI vibration direction is parallel to the long axis, the fibre is described as negative. Between crossed polars, with the 530 nm retardation plate inserted at 45° to the polarizer and analyser vibration directions, the sign of elongation can be determined by observing the colours of fibres that previously had given grey or white first order interference colours between crossed polars. For a retardation plate with the slow direction (usually marked) in the northeast–southwest direction, the first order colours observed are as follows:

Positive fibre	blue–green with fibre northeast–southwest
	orange–yellow with fibre northwest–southeast
Negative fibre	orange–yellow with fibre northeast–southwest
	blue–green with fibre northwest–southeast

Crocidolite is the *only* asbestos type that has a negative sign of elongation. However, exposure to temperatures of about 300 °C or higher may result in a reversal of the sign of elongation of crocidolite to positive. In such cases, however, the thermal history of the fibre is usually indicated by a change of colour.

7.2.3.7.6 Refractive indices

The refractive indices of an asbestos fibre are assessed by mounting a clean separated fibre in a liquid of known RI and orienting it either parallel or perpendicular to the polarizer vibration direction. One or more observations are conducted to determine whether the RI of the fibre is higher than, lower than or equal to that of the immersion liquid.

NOTE Classical mineralogical methods (References [16]–[18]) can be used for determination of refractive indices, but use of these methods requires access to a more extensive range of RI liquids than is specified in this part of ISO 22262, and it is also necessary to prepare multiple slide mounts in order to measure the γ and α indices for asbestos fibres.

Remove all filters from the light path except the daylight colour correction filter and the polarizer. Use the central stop dispersion staining objective to view fibres mounted in a liquid with an RI close to that of the fibre, so that dispersion staining colours can be observed. When dealing with an unknown sample, the observations a) to e) listed in the following can be used to help choose a suitable RI liquid such that the RI of the fibre and the liquid are sufficiently close that dispersion staining colours are produced.

Differences in dispersion between particles and liquids mean that, even though the refractive indices match at one wavelength, they may be quite different at others. This leads to colour effects at the particle/liquid interface when fibres are observed in matching RI liquids using white light. In practice, it is easiest to observe small bright particles and colours against a black background; these conditions are achieved with a central stop in the back focal plane of the objective when used with an axial beam of light produced by the condenser iris. The colours observed at the particle/liquid interface depend on the precise wavelength at which the RI of the liquid and that of the fibres match. When the match of RI is at a wavelength of 589,3 nm (the D line of sodium), the colour at the particle/liquid interface is a deep blue–magenta. For central stop dispersion staining, the colour observed indicates how close, and in which direction, the RI of the particle differs from that of the immersion medium:

a)	Fibre refractive index	>>	Liquid refractive index:	White
b)	Fibre refractive index	>	Liquid refractive index:	Purple–red/orange/yellow
c)	Fibre refractive index	=	Liquid refractive index:	Deep blue–magenta
d)	Fibre refractive index	<	Liquid refractive index:	Blue/blue–green
e)	Fibre refractive index	<<	Liquid refractive index:	White

Different colours are observed when the fibre is oriented parallel or perpendicular to the polarizer vibration direction, arising from the different refractive indices of asbestos fibres in the two perpendicular directions relative to the polarizer vibration direction. A recording of the predominant colours is used to characterize the refractive indices of the fibres. Identification of chrysotile, amosite and crocidolite can be performed with a dispersion staining objective using three high dispersion liquids having the RI values 1,550 for chrysotile, 1,680 for amosite, and 1,700 for crocidolite. In practice, for commercial chrysotile, because of variations in the fibre composition according to the source, a small range of fibre refractive indices and dispersion staining colours may be encountered. The refractive indices of commercial amosite and crocidolite do not vary significantly. For the purpose of this part of ISO 22262, the three RI liquids adequately cover the observed range of refractive indices for chrysotile, amosite, and crocidolite from all known major commercial sources. Crocidolite from Bolivia is an exception in that the refractive indices are lower than those from other sources of crocidolite. However, Bolivian crocidolite is very rare in commerce. Should Bolivian crocidolite be encountered, it can be readily recognized on the basis of its fibrous morphology, negative sign of elongation, and blue–grey pleochroism.

Identification of tremolite, actinolite and anthophyllite can often be performed using a dispersion staining objective using liquids of RI values 1,605 and 1,630. Tremolite or actinolite should be suspected if some of the fibres exhibit oblique extinction, and the γ index observed parallel to the extinction position can be used to define whether the fibre is tremolite or actinolite. If it is important to discriminate between tremolite and actinolite, classify fibres as tremolite if the γ index is estimated to be equal to or lower than 1,637 and as actinolite if the γ index is estimated to be higher than 1,637.

Some sources of talc contain fibres that can be mistaken for anthophyllite. These fibres have intergrowths of both the anthophyllite and talc crystal structures. The fibres exhibit refractive indices that are lower than those of anthophyllite and intermediate between those of talc and anthophyllite. If this type of fibre is present, examine the sample in a liquid of RI 1,615. If no γ indices are observed that are higher than 1,615, classify the fibres as talc. Classify any fibres with γ indices equal to or exceeding 1,615 as anthophyllite.

Identification of richterite/winchite asbestos is difficult by PLM alone. Richterite/winchite should be suspected if the sample also contains vermiculite or talc. Attempts to identify richterite/winchite by PLM alone usually result in classification of the fibres as actinolite, and such an error may be important for regulatory interpretation. Where richterite/winchite is suspected, and the fibres exhibit properties similar to those of actinolite, it is recommended that the fibres be identified by either SEM or TEM.

Annex C shows dispersion staining charts for the α and γ refractive indices of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite, and richterite/winchite in the appropriate RI liquids. Chrysotile exhibits a

small range of refractive indices, depending on the source. For each of the types of asbestos, an acceptable range of colour for the α and γ dispersion staining colours is indicated, representing the observed range in minerals from commercial sources. For chrysotile, it is also important to establish that the λ_0 values for the parallel and normal orientations with respect to the polarizer vibration direction do not differ by more than 100 nm in recognition of its low birefringence. For chrysotile, although there is a range of refractive indices depending on the source, studies have shown that the two indices vary in an approximately parallel manner.

Figures D.1 and D.2 show an example of chrysotile, mounted in 1,550 RI liquid, viewed between crossed polars with the 530 nm retardation plate inserted. Note the fibrillar, wavy appearance, and the blue–green colour in the northeast direction, changing to an orange colour when the fibres are rotated into the northwest direction, showing that the fibres have a positive sign of elongation. Figures D.3 and D.4 show an example of chrysotile viewed under dispersion staining conditions, showing magenta for fibres parallel to the vibration direction of the polarizer and blue for fibres normal to the vibration direction of the polarizer. However, it is necessary to consider that the colours exhibited in the two directions vary depending on the source of the chrysotile and any prior heating or acid treatment. Nevertheless, any variation applies to both the α and γ refractive indices, and the difference between the two (birefringence) remains nearly constant regardless of the source of the chrysotile.

Figures D.5 and D.6 show an example of amosite, mounted in 1,680 RI liquid, viewed between crossed polars with the 530 nm retardation plate inserted. The thin fibres exhibit a blue–green colour in the northeast direction, changing to an orange colour when the fibres are rotated into the northwest direction, showing that the fibres have a positive sign of elongation. Because of the higher birefringence of amosite, some of the thicker fibres exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Figures D.7 and D.8 show amosite viewed under dispersion staining conditions, with a gold colour for fibres parallel to the vibration direction of the polarizer and blue for fibres normal to the vibration direction of the polarizer. Except for heated amosite, these colours vary only slightly for amosite from different sources. The behaviour of heated amosite for the two fibre orientations is illustrated in Figures D.9 and D.10. Heated amosite exhibits significantly higher refractive indices, and dark brown–light brown pleochroism for fibres parallel and normal to the polarizer vibration directions, respectively.

Figures D.11 and D.12 show an example of crocidolite, mounted in 1,700 RI liquid, viewed between crossed polars with the 530 nm retardation plate inserted. The fibres exhibit a yellow–orange colour in the northeast direction, changing to a blue colour when the fibres are rotated into the northwest direction, showing that the fibres have a negative sign of elongation. The birefringence of crocidolite is very low, so the dispersion staining colours for fibres parallel and normal to the polarizer vibration direction are not very different. However, a lighter blue is discernable for the parallel direction, indicating that the lower RI is parallel to the length of the fibre (Figures D.13 and D.14). The blue–grey pleochroism of crocidolite is shown in Figures D.15 and D.16. The behaviour of heated crocidolite for the two fibre orientations is illustrated in Figures D.17 and D.18. Heated crocidolite exhibits dark brown–light brown pleochroism for fibres parallel and normal to the polarizer vibration directions, respectively. For heated crocidolite such as that illustrated, the sign of elongation is positive, and in this condition electron microscopy with energy dispersive X-ray analysis is necessary to discriminate between crocidolite and amosite.

Figures D.19 and D.20 show an example of SRM 1867 tremolite, mounted in 1,605 RI liquid, viewed between crossed polars with the 530 nm retardation plate inserted. The thin fibres exhibit a blue–green colour in the northeast direction, changing to an orange colour when the fibres are rotated into the northwest direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence of tremolite, some of the thicker fibres can exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Figures D.21 and D.22 show SRM 1867 tremolite viewed under dispersion staining conditions, with a yellow colour for fibres parallel to the extinction position closest to the vibration direction of the polarizer and dark blue for fibres at the other extinction position. The dark blue colour of the fibre in Figure D.22 and the magnitude of the extinction angle indicate that this fibre presents the α RI at this orientation. Figures D.23 to D.26 show SRM 1867 tremolite mounted in 1,625 RI liquid, which is intermediate between the γ and α indices of the fibres. Figures D.35 to D.38 show an example of HSE reference tremolite, mounted in 1,605 RI liquid. This variety of tremolite exhibits parallel extinction.

Figures D.27 and D.28 show an example of SRM 1867 actinolite, mounted in 1,630 RI index liquid, viewed between crossed polars with the 530 nm retardation plate inserted. The thin fibres exhibit a blue–green colour in the northeast direction, changing to an orange colour when the fibres are rotated into the northwest direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence of tremolite, some of the thicker fibres can exhibit first and second order interference colours that can be compared with the

interference colour chart in Annex B. Figures D.29 and D.30 show SRM 1867 tremolite viewed under dispersion staining conditions, with a purple–red colour for fibres parallel to the extinction position closest to the vibration direction of the polarizer and light blue for fibres at the other extinction position. Figures D.39 to D.44 show an example of HSE reference actinolite mounted in 1,640 RI liquid. The HSE actinolite is considerably more asbestiform than the SRM 1867 actinolite, and exhibits parallel extinction as well as pleochroism as illustrated in Figures D.43 and D.44.

Figures D.31 and D.32 show an example of SRM 1867 anthophyllite, mounted in 1,605 RI liquid, viewed between crossed polars with the 530 nm retardation plate inserted. The thin fibres exhibit a blue–green colour in the northeast direction, changing to an orange colour when the fibres are rotated into the northwest direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence of anthophyllite, some of the thicker fibres can exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Figures D.33 and D.34 show anthophyllite viewed under dispersion staining conditions, with blue–purple colours for fibres parallel to the vibration direction of the polarizer and light blue for fibres normal to the vibration direction of the polarizer. Figure D.33 shows some fibres that exhibit purple dispersion staining colours. This indicates that the RI in that orientation is higher than 1,630, representing the γ index. Other fibres exhibit a blue colour, which indicates that the RI in the particular axial orientation is lower than 1,630. This is probably a result of intergrowths of talc in the fibre bundle, since all fibres in this orientation relative to the polarizer direction should exhibit only the γ index. Figures D.45 to D.48 show an example of HSE reference anthophyllite in 1,605 RI liquid.

Figures D.49 and D.50 show an example of richterite/winchite, mounted in 1,630 RI liquid, viewed between crossed polars with the 530 nm retardation plate inserted. The thin fibres exhibit a blue–green colour in the northeast direction, changing to an orange colour when the fibres are rotated into the northwest direction, showing that the fibres have a positive sign of elongation. Because of the moderate birefringence, some of the thicker fibres exhibit first and second order interference colours that can be compared with the interference colour chart in Annex B. Figures D.51 and D.52 show richterite/winchite viewed under dispersion staining conditions, with a purple colour for fibres parallel to the extinction position closest to the vibration direction of the polarizer and blue for fibres at the other extinction position. Regardless of the highly asbestiform appearance of this sample, the fibres exhibit oblique extinction.

7.2.4 Interferences

7.2.4.1 Heated asbestos

Changes occur to asbestos when it is heated. Therefore, care should be taken if sample preparation involves heating the asbestos-containing material. Even short exposure of crocidolite to temperatures of 300 °C to 500 °C may cause colour changes, and increases in both RI and the birefringence. For crocidolite, the changes with heating are: the sign of elongation reverses and the colour changes from grey to yellow then orange–brown; pleochroism is suppressed at the grey coloration stage, but reappears on further heating. For amosite, the sign of elongation remains positive, but the colour changes from yellow to a dark brown, and pleochroism is observed. Thus, heat-degraded crocidolite and amosite cannot be distinguished from each other by light microscopy after exposure to temperatures above about 500 °C. The refractive indices of chrysotile increase after significant exposure to temperatures of about 600 °C or greater: the birefringence decreases and, in a few cases, the sign of elongation changes to negative and the fibres become pale brown. The alteration of asbestos by heat is dependent on both the duration and the temperature of exposure. Prolonged exposure to high temperatures can result in complete degradation, but, with judicious sampling, unaffected fibres can often be detected in peripheral locations or in debris that became detached during installation. However, in extreme situations, analytical electron microscopy may be required to aid identification. Examples of heated amosite and crocidolite in plane polarized light are shown in Annex D.

7.2.4.2 Leached chrysotile

Exposure of chrysotile to acidic aqueous media may result in reduction of the refractive indices as a consequence of leaching of magnesium from the crystal structure. Progressive leaching also results in reduction of the birefringence, and ultimately the fibre becomes isotropic. In addition to the action of mineral acids used in some of the procedures in this part of ISO 22262, leaching may also occur in chrysotile exposed to aggressive water (water with only low mass fractions of dissolved calcium and magnesium, and with low pH values). Leached

chrysotile may be encountered on the surfaces of chrysotile cement products such as roofing materials after long periods of exposure to rain.

7.2.4.3 Fibres with morphological and/or optical properties similar to those of asbestos

Most of the fibres discussed in the following paragraphs occur infrequently in samples presented for analysis. However, analysts need to be aware of their existence and distinguishing characteristics in PLM. There are five types of fibre that can resemble chrysotile. Some mineral fibres can also superficially resemble amphiboles.

Polyethylene is the most important of the interfering fibres because it is used as an asbestos substitute. Shredded polyethylene resembles chrysotile. In 1,550 RI liquid, the dispersion staining colours are within the range for those of chrysotile, although experienced analysts will observe morphological differences and de-saturation of the blue colour perpendicular to the fibres because of the low RI in this direction. The birefringence is also higher than that of chrysotile. If polyethylene is suspected, the melting of fibres on a hot plate or in a flame will readily distinguish them from chrysotile.

Fibres from leather have low birefringence and similar dispersion staining colours to chrysotile. At magnifications below 100 times, they appear to have similar morphology to that of chrysotile, but they usually exhibit clearly visible uniform fibrils. Individual chrysotile fibrils are too small to be seen by PLM, although uniform bundles of fibrils are visible. In most instances, the differences between chrysotile and leather can be detected during stereomicroscope examination. If leather is suspected to be present, the sample may be ashed at 400 °C to remove it, and then the residual ash can be reexamined for identification of asbestos. Care should be taken not to allow the sample temperature to rise above 500 °C.

Macerated aramid fibres may appear to have a morphology similar to that of chrysotile, but these fibres can be recognized by their extremely high birefringence showing high-order white interference colours. When mounted in 1,550 RI liquid, the refractive indices are clearly inconsistent with those of chrysotile.

Spider web and natural organic fibres such as cellulose and feathers have refractive indices close to those of chrysotile and show similar interference colours between crossed polars. In a sample with little non-fibrous material, the morphology of these fibres can be readily distinguished from that of chrysotile. However, in samples containing significant particulate material, sometimes only a small portion of the fibre can be observed due to obscuration by the particles and this can lead to misidentification. These fibres can be removed by ashing the sample or exposing individual fibres to a flame.

Talc fibres are thin ribbons that may sometimes be recognized by characteristic morphological twists. For the RI parallel to the fibre length, they have a value in the range 1,589 to 1,600, resulting in a pale yellow dispersion staining colour when immersed in 1,550 RI liquid. The other two refractive indices of talc are in the ranges 1,539 to 1,550 and 1,589 to 1,600, and with a dispersion staining objective, blue and pale yellow colours perpendicular to the fibre are observed in 1,550 RI liquid at different orientations as the fibre is “rolled”. It is important to demonstrate that the γ index of any straight fibres that do not exhibit ribbon-like morphology is lower than 1,615, in order to exclude the possibility that the fibres are anthophyllite

Fibrous brucite normally consists of straight white to pale brown fibres, but brucite lacks the tensile strength of asbestos. It is brittle and is soluble in acid. Brucite has a negative sign of elongation, which reverses to positive when heated. Sometimes brucite fibres may appear to be isotropic. It is distinguished from chrysotile by its refractive indices. In central stop dispersion staining, brucite yields colours of yellow to pale yellow in 1,550 RI liquid.

Superficially, fibrous wollastonite can be mistaken for tremolite. Fibrous wollastonite has an acicular morphology, is very brittle, white in appearance, and is slowly soluble in acid. After treatment for a short time (e.g. 15 min) in 100 g/l hydrochloric acid, the fibres exhibit etched areas. Wollastonite always displays a non-zero extinction angle. The RI almost parallel to the fibre is in the range 1,628 to 1,650. The other two refractive indices are in the ranges 1,626 to 1,640, and 1,631 to 1,653, and are observed across the fibre, at different orientations as the fibre is rolled. A distinctive feature is that the RI with the length of the fibre almost parallel to the polarizer vibration direction is intermediate between the two refractive indices observed at the different orientations across the fibre as the fibre is rolled. Examination of many fibres with crossed polars and with the 530 nm retardation plate inserted shows most as having a positive sign of elongation, and fibres in other orientations appear to have a negative sign of elongation. Gentle pressure on the cover slip with a needle can be used to rotate a fibre and show it to change from a positive to a negative sign of elongation as it is rolled into a different axial orientation.

Diatomaceous earth may exhibit acicular fragments with the appearance of fibres. However, these fibres have a low RI of approximately 1,42 and are readily distinguished from asbestos fibres using dispersion staining. Also, there is usually characteristic morphology that can be recognized when the material is examined at magnifications around 500 times.

7.2.4.4 Identification of other sample components

A laboratory conducting routine analysis selectively removes fibres for examination and ignores the majority of the non-asbestos materials. The composition of many asbestos products is relatively uniform during the manufacture and a wider knowledge of these non-asbestos materials can be helpful in recognizing many common products or formulations. Because of this, the analyst should become familiar with the information in Annex A.

8 Analysis by SEM

8.1 General

Complete details relating to identification of mineral fibres, including asbestos fibres, using SEM are given in ISO 14966.[7]

8.2 Requirements

8.2.1 Scanning electron microscope, with an accelerating voltage of at least 20 kV.

8.2.2 Energy dispersive X-ray system. The SEM shall be equipped with an energy dispersive X-ray analyser capable of achieving a resolution better than 170 eV (FWHM) on the Mn K_α peak. The performance of an individual combination of SEM and solid-state X-ray detector is dependent on a number of geometrical factors. The X-ray detector shall be capable of detecting sodium in crocidolite, in order to permit discrimination between crocidolite and amosite.

8.2.3 Vacuum coating unit, capable of producing a vacuum better than 0,013 Pa. It shall be used for vacuum deposition of carbon on the SEM specimens. A sample holder is required which allows the SEM specimens to be continuously rotated and tilted during the coating procedure.

8.3 Calibration

For the purposes of this method, calibration consists of obtaining EDXA spectra from reference samples of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite, and richterite/winchite. The chemical compositions of commercial chrysotile, amosite, crocidolite and anthophyllite do not vary substantially, and comparison of unknown EDXA spectra with those from the three reference asbestos samples constitutes sufficient identification for this part of ISO 22262. For most purposes, it is not necessary to discriminate between tremolite and actinolite, since the compositional boundary between them is a matter of convention. When it is necessary to discriminate between tremolite and actinolite, the SRM 1867 tremolite and actinolite samples are particularly useful, since these samples have compositions just below and just above the boundary defined by the International Mineralogical Association. In some applications, the magnesium may be partially leached from chrysotile, leading to a chemical composition that approaches that of talc. In order to facilitate the discrimination between chrysotile and talc or anthophyllite, it is recommended that an EDXA spectrum also be obtained from a known sample of talc. Use this spectrum to define the upper limit of the magnesium mass fraction in talc. Examples of EDXA spectra obtained on the SRM 1866 and SRM 1867 samples, the HSE reference asbestos samples, Bolivian crocidolite and richterite/winchite are illustrated in Annex E. For positive identification, reference EDXA spectra from asbestos standards similar to those shown in Annex E should be recorded using the specific combination of SEM and EDXA detector, since the geometries and detector efficiencies vary between different instruments.

8.4 Sample preparation

Select representative fibres, either from the original laboratory sample or from the residue remaining after treatment according to the procedures specified in 7.2.2 and 7.2.3. Mount these fibres either directly on a graphite SEM stub or on double-sided adhesive tape on an SEM stub. Place the SEM stub in the vacuum coating unit and evaporate a thin film of carbon on to the surface of the fibres.

8.5 Qualitative analysis by SEM

8.5.1 Acquisition of EDXA spectra

It is important to obtain the EDXA spectrum from clean areas of the fibre, since distortion of peak heights by contributions from attached particles may compromise the identification. Particles adjacent to the fibre under analysis may also contribute to the EDXA spectrum, and this effect should be minimized to the extent possible.

8.5.2 Sample analysis

The SEM stub with the unknown fibres is examined at a low magnification in the SEM, and EDXA spectra are acquired from regions of the fibres that are clear of other attached particles. The EDXA spectra are compared with the reference spectra.

8.5.2.1 Chrysotile

Classify a fibre as chrysotile if:

a) the Mg and Si peaks are clear, and comparable in Mg/Si peak height ratio with that of the reference;

b) any Fe, Mn and Al peaks are small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

IMPORTANT — Anthophyllite and talc both yield EDXA spectra that conform to these specifications, but the Mg/Si peak height ratio for these minerals is lower than that for chrysotile. In order to avoid erroneous classification of talc or anthophyllite as chrysotile, take account of the Mg/Si peak height ratio and calibrate the EDXA detector using known samples of chrysotile and talc.

8.5.2.2 Amosite

Classify a fibre as amosite if:

a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference amosite;

b) no statistically significant peaks from Na or Al are present;

c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

8.5.2.3 Crocidolite

Classify a fibre as crocidolite if:

a) the Na, Si and Fe peaks are comparable in ratio with those of the reference crocidolite;

b) any peak from Mg is small, and no peaks from Al or Mn are visible.

NOTE 1 Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

NOTE 2 If a large peak from Mg is present, it is possible that the fibre is magnesio-riebeckite. Bolivian crocidolite is the only known commercial source, although this variety of crocidolite can occur as contamination of other minerals.

8.5.2.4 Tremolite

Classify a fibre as tremolite if:

a) the Mg, Si, Ca and Fe peaks are comparable in ratio to those of reference tremolite;

b) no statistically significant peaks from Na or Al are present;

c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

8.5.2.5 Actinolite

Classify a fibre as actinolite if:

a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference actinolite;

b) no statistically significant peaks from Na or Al are present;

c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

8.5.2.6 Anthophyllite

Classify a fibre as anthophyllite if:

a) the fibre is straight and exhibits no evidence of a ribbon-like structure;

b) the Mg and Si peaks are comparable in ratio to those of the reference anthophyllite — anthophyllite from some sources may not exhibit a peak from Fe, although in commercial anthophyllite a peak from Fe will probably be observed;

c) no statistically significant peaks from Na or Al are present;

d) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

8.5.2.7 Sodic–calcic amphibole asbestos (richterite/winchite)

Classify a fibre as sodic–calcic amphibole if:

a) the spectrum is similar to that of actinolite or tremolite, but the Ca peak is substantially smaller and an Na peak is present — a K peak may also be evident;

b) no statistically significant peak from Al is present;

c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

9 Analysis by transmission electron microscope

9.1 General

Full details relating to identification of asbestos fibres using TEM are given in ISO 10312[2] and ISO 13794[4]. Additional information on the investigation of minerals using TEM is given in References [26]–[29]. A simple technique for quantitative measurement of electron diffraction patterns is available (Reference [30]). Detailed interpretation of single-crystal electron diffraction patterns, sometimes required for definitive identification of amphibole fibres, can be accomplished using a computer program, e.g. XIDENT (Reference [31]).

9.2 Requirements

9.2.1 Transmission electron microscope, operating at an accelerating potential of 80 kV to 120 kV. The TEM shall have an illumination and condenser lens system capable of forming an electron probe smaller than 250 nm in diameter.

9.2.2 Energy dispersive X-ray analyser. The TEM shall be equipped with an energy dispersive X-ray analyser capable of achieving a resolution better than 170 eV (FWHM) on the Mn K_α peak. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of geometrical factors, the required performance of the combination of the TEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid-state X-ray detectors are least sensitive in the low-energy region, and so measurement of sodium in crocidolite is the primary performance criterion. The combination of electron microscope and X-ray analyser shall yield, under routine analytical conditions, a peak from sodium that allows discrimination between the spectra from crocidolite and amosite.

9.2.3 Vacuum coating unit. If carbon-coated specimen grids are not available, a vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon for preparation of carbon-coated grids.

9.2.4 Calibration grids. TEM specimen grids prepared from dispersions of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite, richterite/winchite, and talc are required for calibration of the EDXA system. It is recommended that gold or nickel grids be used to facilitate detection of sodium. For calibration of the camera constant for interpretation of ED patterns, TEM specimen grids with vacuum-evaporated thin films of gold, aluminium or thallous [Tl(I)] chloride deposited on to carbon films are required.

9.2.5 Disposable tip micropipettes, suitable for transferring a volume of approximately 3 µl to a carbon-coated TEM specimen grid.

9.3 Calibration

9.3.1 EDXA system

For the purposes of this method, calibration consists of obtaining EDXA spectra from reference samples of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite, and richterite/winchite. The chemical compositions of commercial chrysotile, amosite, crocidolite, and anthophyllite do not vary substantially, and comparison of unknown EDXA spectra with those from the three reference asbestos samples constitutes sufficient identification for this part 1 of ISO 22262. For most purposes, it is not necessary to discriminate between tremolite and actinolite, since the compositional boundary between them is a matter of convention. When it is necessary to discriminate between tremolite and actinolite, the SRM 1867 tremolite and actinolite samples are particularly useful since they have compositions just below and just above the boundary defined by the International Mineralogical Association (Reference [24]). In some applications, the magnesium may be partially leached from chrysotile, leading to a chemical composition that approaches that of talc. In order to facilitate the discrimination between chrysotile and talc or anthophyllite, it is recommended that an EDXA spectrum also be obtained from a known sample of talc. Use this spectrum to define the upper limit of the magnesium mass fraction in talc. Examples of EDXA spectra obtained on the SRM 1866 and SRM 1867 samples, the HSE reference asbestos samples, Bolivian crocidolite, and richterite/winchite appear in Annex F. For positive identification, reference EDXA spectra from asbestos standards similar to those shown in Annex F should be recorded using the specific combination of TEM and EDXA detector, since the geometries and detector efficiencies vary between different instruments.

9.3.2 Camera constant for interpretation of ED patterns

Use gold, aluminium or thallous [Tl(I)] chloride to calibrate the radius-based camera constant, λL, the product of the wavelength and camera length, for electron diffraction patterns. Specimen grids with a vacuum deposited, thin, polycrystalline film of one of these materials on a thin carbon film are used for the calibration. The calibration data for the first two diffraction rings, where D is the ring diameter, are shown in Table 6.

Table 6 — Radius-based camera constants

Calibration material	Radius-based camera constant λL	
	1st diffraction ring	2nd diffraction ring
Gold	0,117 74D	0,101 97D
Aluminium	0,116 90D	0,101 24D
Thallous [Tl(I)] chloride	0,192 14D	0,135 86D

9.4 Sample preparation

Remove representative fibres from the sample (see 7.2.2 and 7.2.3), and place them in an agate mortar, and pestle. Add approximately 1 ml of ethanol, and grind the fibres with the pestle until they are well dispersed in the ethanol. Set up a laboratory stand and clamp, and use it to hold a pair of fine-point tweezers that are supporting a carbon-coated TEM specimen grid, with the carbon side facing upwards. Using a disposable tip micropipette, drop a 3 µl volume of the ethanol dispersion on to the grid, and allow it to dry. Drying is faster if the grid is held under a heat lamp. When dry, the TEM grid is ready for examination.

If crocidolite or sodic–calcic amphibole is suspected, use of a carbon-coated gold TEM grid is recommended in order to avoid partial overlap of the Na K_α peak by the Cu L_α X-ray peak if a copper grid is used.

9.5 Qualitative analysis by TEM

9.5.1 Acquisition of EDXA spectra

It is important to obtain the EDXA spectrum from clean areas of the fibre, since distortion of peak heights by contributions from attached particles may compromise the identification.

9.5.2 Chrysotile

The morphological structure of chrysotile as seen in the TEM is characteristic and, with experience, can be recognized readily. However, a few other minerals have a similar appearance, and morphological observation by itself is inadequate for most samples.

Classify a fibre as chrysotile if:

a) the Mg and Si peaks are clear, and comparable in Mg/Si peak height ratio with that of reference chrysotile;

b) any Fe, Mn and Al peaks are small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

IMPORTANT — Anthophyllite and talc both yield EDXA spectra that conform to these specifications, but the Mg/Si peak height ratio for these minerals is lower than that for chrysotile. In order to avoid erroneous classification of talc or anthophyllite as chrysotile, take account of the Mg/Si peak height ratio and calibrate the EDXA detector using known samples of chrysotile and talc.

9.5.3 Amosite

Classify a fibre as amosite if:

a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference amosite;

b) no statistically significant peaks from Na or Al are present;

c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

9.5.4 Crocidolite

Classify a fibre as crocidolite if:

a) the Na, Si and Fe peaks are comparable in ratio with those of the reference crocidolite;

b) no statistically significant peak from Al is present;

c) any peak from Mg is small, and no Mn peak is visible.

NOTE 1 Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

NOTE 2 If a large peak from Mg is present, it is possible that the fibre is magnesio-riebeckite. Bolivian crocidolite is the only known commercial source, although this variety of crocidolite can occur as contamination of other minerals.

9.5.5 Tremolite

Classify a fibre as tremolite if:

a) the Mg, Ca and Fe peaks are comparable in ratio with those of the reference tremolite;

b) no statistically significant peak from Al is present;

c) any peak from either Na or K is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

9.5.6 Actinolite

Classify a fibre as actinolite if:

a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference actinolite;

b) no statistically significant peaks from Na or Al are present;

c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

9.5.7 Anthophyllite

Classify a fibre as anthophyllite if:

a) the fibre is straight and exhibits no evidence of a ribbon-like structure;

b) the Mg and Si peaks are comparable in ratio to those of reference anthophyllite — anthophyllite from some sources may not exhibit a peak from Fe, although in commercial anthophyllite a peak from Fe will probably be observed;

c) no statistically significant peaks from Na or Al are present;

d) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

9.5.8 Sodic–calcic amphibole asbestos (richterite/winchite)

Classify a fibre as sodic–calcic amphibole if:

a) the spectrum is similar to that of actinolite or tremolite, but the Ca peak is substantially smaller and an Na peak is present — a K peak may also be evident;

b) no statistically significant peak from Al is present;

c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

10 Test report

The test report shall contain at least the following information:

a) reference to this part of ISO 22262 (ISO 22262-1:2012);

b) the identification of the sample, including the location (if known by the analyst);

c) the date of the analysis;

d) the identity of the analyst;

e) all applicable specimen preparation details;

f) any procedure used not specified in this part of ISO 22262 or regarded as an optional procedure;

g) the variety or varieties of asbestos detected;

h) the analytical method used to identify the asbestos.

Items i) to k) shall be recorded in the laboratory data, but the extent to which they are included as part of the test report is optional:

i) the observations made to confirm the identification of the asbestos varieties reported, including any optional procedures;

j) the estimated mass fraction(s) of the asbestos varieties detected in ranges as follows:

   1) none detected,

   2) detected,

   3) 0,1 % to 5 %,

   4) 5 % to 50 %,

   5) 50 % to 100 %;

NOTE 1 These categories for reporting asbestos mass fractions are estimates only; they are intended to provide guidance in the interpretation of results. If it is necessary to make critical decisions on the basis of results in the range from “non-detected” to 5 %, sample analysis by a quantitative method is appropriate (e.g. using ISO 22262-2).

NOTE 2 The reporting category “detected” provides the analyst with a means of reporting the result when only one or two fibres are detected in the analysis, the observation of which may be a consequence of unintended contamination of the sample.

k) the variety or varieties of any non-asbestos fibres detected, and the observations made which allowed these fibres to be discriminated from asbestos fibres.

An example of a suitable format for the test report is shown in Annex H.

Annex A
(normative)

Types of commercial asbestos-containing material

The properties of asbestos such as non-flammability, chemical stability, and high strength have led worldwide to a broad use of this mineral in the building and industrial sectors. Asbestos–cement products, asbestos-containing lightweight panels and fire-prevention panels, asbestos packings and asbestos cloths, asbestos boards, asbestos foams, asbestos-containing fireproofing and acoustic and decorative plasters (sprayed asbestos), and asbestos-containing compositions for trowel application and putties are the most important uses. In addition, there is also a variety of products to which asbestos fibres were frequently added at smaller mass fractions, e.g. paints for protective coatings, adhesives, plastic sheets, and tiles.

Table A.1 gives the most important asbestos-containing materials with examples of their applications and the typical asbestos mass fractions. In exceptional cases, asbestos mass fractions deviating from those quoted may have been used.

Table A.1 — Asbestos-containing materials; examples of use and typical asbestos content

Product	Examples of application	Typical asbestos type and mass fraction
Asbestos–cement flat boards	Roof claddings Sidings Banister elements Windowsills Staircases Partition walls Support for cable runs In small sizes as slates and shingles in the roofing and siding sectors	Chrysotile 10 %–12 %, Sometimes also <5 % crocidolite or amosite in addition to chrysotile
Asbestos–cement corrugated sheets	Roof claddings Perimeter insulation Sidings in the industrial sector	Chrysotile 10 %–12 %, sometimes also, with some manufacturers, <5 % crocidolite in addition to chrysotile
Asbestos–cement pipes or ducts	Drinking water and wastewater pipes Service pipes Inlet air and exhaust air ducts Cable shafts	Chrysotile 10 %–15 %. Drinking water pipe also <5 % crocidolite or amosite in addition to chrysotile
Asbestos–cement mouldings	Standard ashtrays Flower boxes Garden articles Sculptures	Chrysotile 10 %–12 %

Table A.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction
Asbestos-containing lightweight building boards or fire-resistant panels	Sealing of openings in walls required to be fire resistant Fire-protection encasement of ventilation ducts, cable ducts and cable shafts Fire closures in walls required to be fire resistant (fire shutters, fire barriers) Fire-protection encasements Smoke-removal ducts Insert in fire-resistant doors and gates Substructure of luminaries (lighting fixtures)	Chrysotile ~15 % and amosite ~15 %
Asbestos-containing lightweight building boards or fire-resistant panels	Lining fire-hazard rooms Partition walls, partition surfaces, doors Sanitary modules Support and beam encasements Smoke aprons Fire locks	Chrysotile <50 %, sometimes amosite <35 %
Asbestos-containing pipe and boiler insulations	Corrugated paper pipe insulation 85 % magnesia block and pipe insulation Calcium silicate block and pipe insulation	Chrysotile 30 %–100 % Total of 15 % asbestos, can be chrysotile, amosite or crocidolite, or any mixture of two or more.
Asbestos packing, asbestos cloth	Seals or sealing strips on lightweight walls required to be fire resistant (at ceiling, floor, joints between elements, wall terminations) Seals on pipe and duct feed-throughs in walls and ceilings Seals between flanges of ventilation ducts Seals on fire-resistant glazing, shelter doors, chimney soot doors Seals and insulation on heat-generation systems, hot pipes and hot valves Fire blankets Heat-resistant clothing, heat-resistant gloves Lining of pipe clips for hot water, steam and sprinkler pipes Lamp wicks Mantles for gas lamps	Predominantly chrysotile (80 %–100 %); crocidolite for acid-resistant applications
Asbestos millboards	Sealing strips on lightweight walls required to be fire resistant (at ceiling, floor, joints between elements, wall terminations) Substructure of luminaries (lighting fixtures) Bottom coating of wooden windowsills over radiators	Chrysotile 80 %–100 %
Asbestos foams	Infilling (sealing) of movement joints Seals at fire shutters and fire barriers	Chrysotile ~50 %

Table A.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction
Sprayed asbestos	Contour-following fire-resistant coating of steel structures Coating of ceilings and walls in music auditoria, theatres, churches, garages, industrial rooms (for noise protection) Sealing off openings for cable, pipe and duct feed-throughs through walls required to be fire resistant Encasing of ventilation ducts	Chrysotile, crocidolite or amosite 40 %–70 %, also mixtures of mineral wool with either 20 % amosite or up to 30 % chrysotile. Other mixtures include 15 % chrysotile with either perlite or vermiculite, and gypsum. Sprayed vermiculite coatings (with or without chrysotile) can contain up to 2 % tremolite, some of which can be asbestiform. Several per cent of tremolite asbestos (Japan)
Sprayed decorative coatings (texture coats)	Coating of ceilings and walls to provide a textured surface which masks irregularities	Chrysotile <5 %. Some constituents can also contain tremolite. Some of the tremolite can be asbestiform.
Gypsum wallboard joint compounds	Provides smooth joint between adjacent panels	Chrysotile <5 %. Some constituents can also contain low mass fractions of tremolite.
Asbestos-containing troweled-on compositions and putty	Grouting of prefabricated concrete components Sealing of movement joints Pipe feeds through walls and ceilings Door casings of fire-resistant doors Anti-drumming coatings (car preservation) Coating of underwater structures Baseboard coating on house walls	Chrysotile <20 %
Asbestos-containing floorings	Reinforcement in flexible sheets Rot-resistant support layer as underlay of cushion PVC flooring materials	Chrysotile 10 %–20 % Chrysotile 80 %–100 %
Asphalt or PVC asbestos floor tiles	Reinforcement	Asphalt tiles containing chrysotile <35 %, PVC tiles containing chrysotile <20 %
Rubberized asbestos seals	Gaskets for pipe flanges	Chrysotile 50 %–90 %
Asbestos-containing friction products	Brake linings Brake bands Clutch linings	Chrysotile 10 %–70 %
Acid-resistant containers	Lead-acid battery boxes Drums for acid	Crocidolite 10 %–50 %
Filter media	Air filters Liquid filters Sterile and aseptic filters Clarifying sheets Diaphragms for chloralkali electrolysis processes Filtration media for Gooch crucibles	Chrysotile, rarely amosite 95 % For Gooch crucibles, 100 % tremolite or anthophyllite

Table A.1 *(continued)*

Product	Examples of application	Typical asbestos type and mass fraction
Talc (asbestos content dependent on deposit)	Release agents for electric cables, rubber products Release agents in the confectionery industry Tailor's chalk Paper manufacture Medicine, cosmetics	Chrysotile and/or actinolite/ tremolite. Some of the actinolite/ tremolite can be asbestiform
Vermiculite (exfoliated)	Attic and wall cavity insulation Fireproofing Horticultural products	Depends on the source of the vermiculite. Vermiculite from Montana, USA, can contain up to 6 % of a mixture of amphibole types, some of which can be asbestiform
Industrial minerals: wollastonite, sepiolite, attapulgite	Ceramics manufacture Plastics fillers Surfacing materials and joint compounds Ceiling tiles Drilling muds (attapulgite)	Depends on the source of the mineral. Can contain several per cent of tremolite or actinolite, some of which can be asbestiform.
Industrial minerals: calcite, dolomite and gypsum	Manufacture of building materials Industrial uses	Depends on the source of the mineral. Carbonate minerals can contain several per cent of tremolite or actinolite, some of which can be asbestiform
Industrial minerals: mica	Ceramics manufacture Manufacture of building materials	Depends on the source of the mineral. Can contain tremolite or actinolite, some of which can be asbestiform
Asphalt surfacings	Road construction	Chrysotile, generally ≤1 %
Wall and ceiling plasters	Interior wall and ceiling coatings, with or without aggregate and fibres such as animal hair or jute	Chrysotile. Generally locally mixed and inhomogeneous. Can be any mass fraction up to approximately 3 %
Drilling muds	Oil exploration, rock drilling	Chrysotile. Often the chrysotile is very fine and short, sometimes originating from Coalinga, California. Can contain <100 % chrysotile
Chemical products for construction, and other products	Bitumen, roofing and sealing sheets	Chrysotile <30 %
	Sealing putties	Chrysotile <2 %
	Glazing putties	Chrysotile <4 %
	Bituminous coatings	Chrysotile <30 %
	Fillers and sealers	Chrysotile <25 %
	Jointing compounds	Chrysotile <5 %
	Paints	Chrysotile <9 %
	Glues	Chrysotile <4 %
	Fire retardants	Chrysotile <10 %
	Sub-floor protection	Chrysotile <4 %

Annex B
(normative)

Interference colour chart

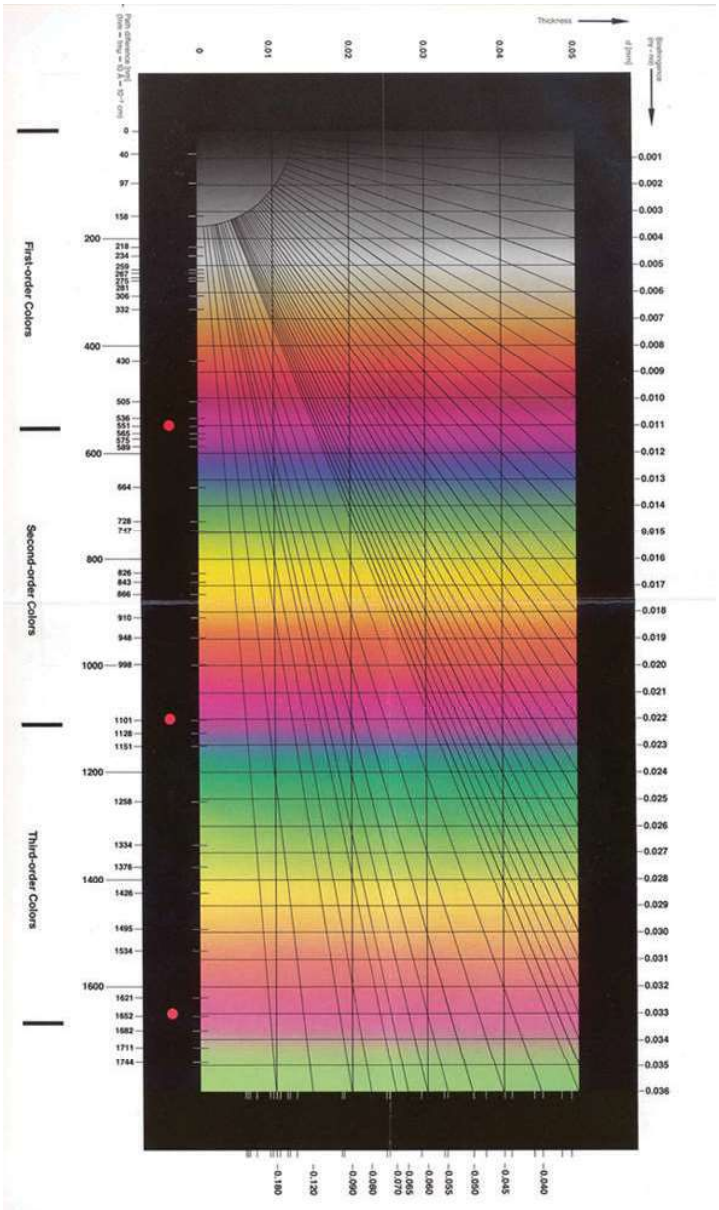


Figure B.1 — Interference colour chart

Annex C
(normative)

Dispersion staining charts

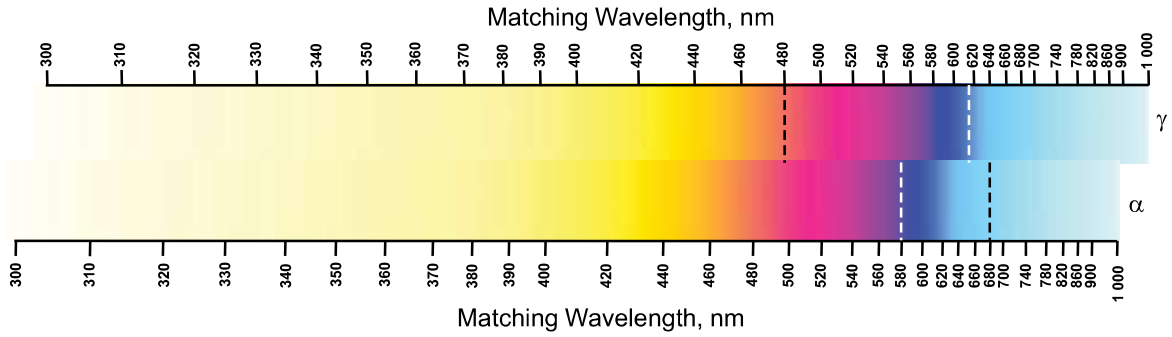


Figure C.1 — Central stop dispersion staining colours for chrysotile in 1,550 RI liquid

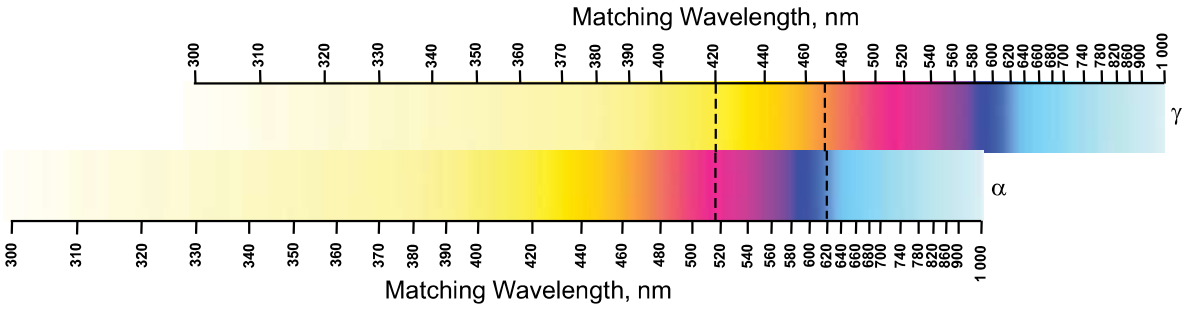


Figure C.2 — Central stop dispersion staining colours for amosite in 1,680 RI liquid

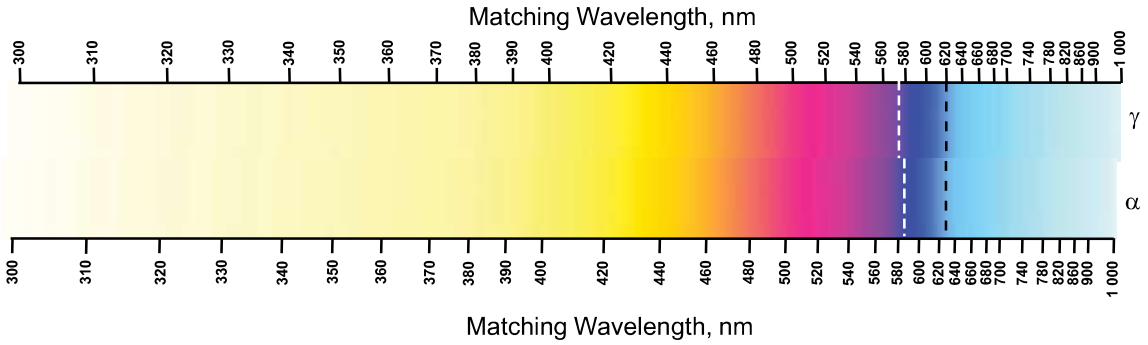


Figure C.3 — Central stop dispersion staining colours for crocidolite in 1,700 RI liquid

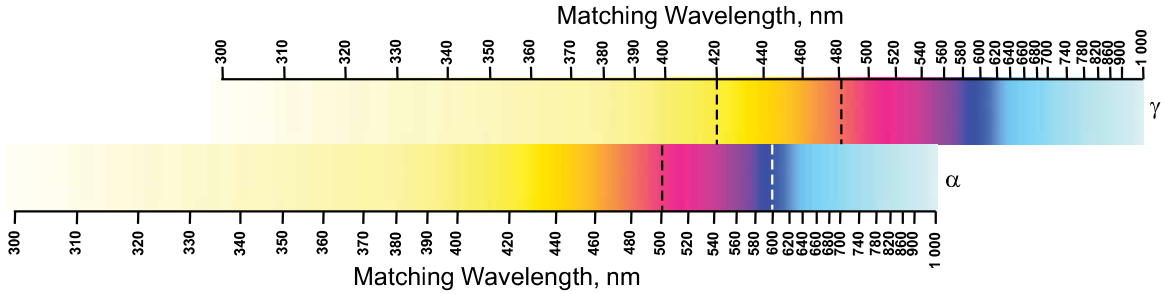


Figure C.4 — Central stop dispersion staining colours for tremolite in 1,605 RI liquid

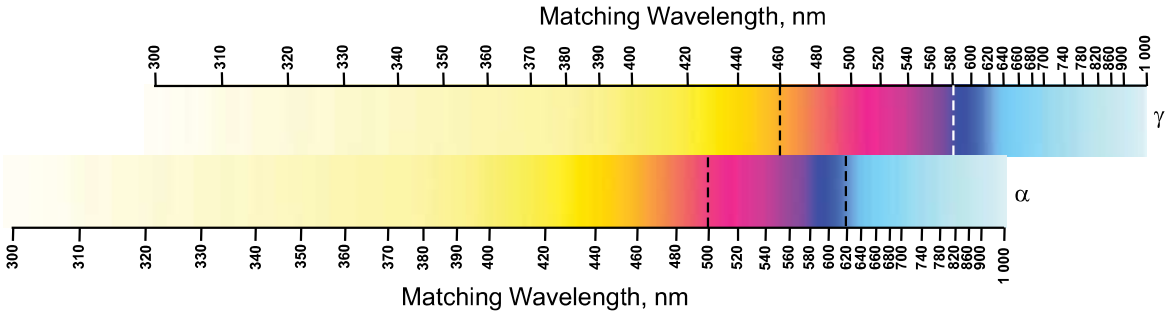


Figure C.5 — Central stop dispersion staining colours for actinolite in 1,630 RI liquid

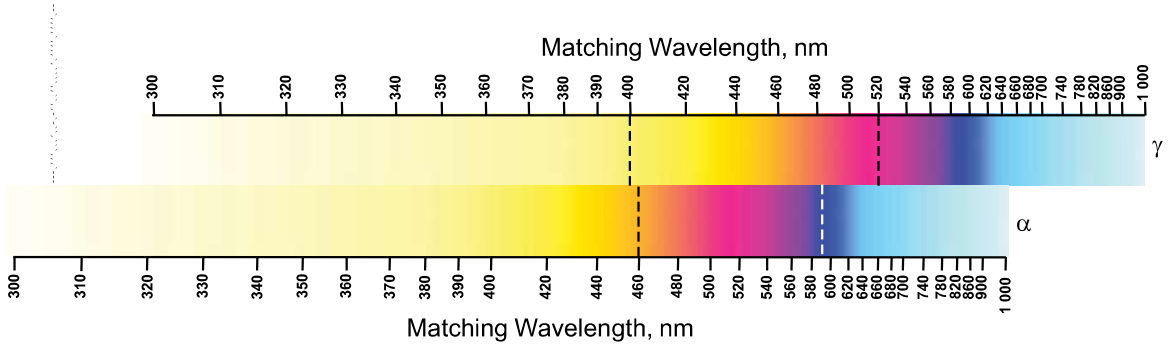


Figure C.6 — Central stop dispersion staining colours for anthophyllite in 1,605 RI liquid

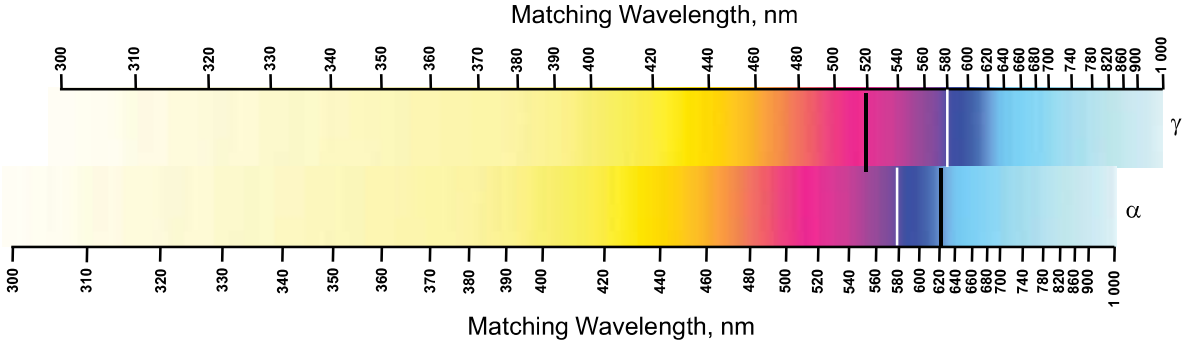


Figure C.7 — Central stop dispersion staining colours for richterite/winchite asbestos in 1,630 RI liquid

Annex D
(normative)

Asbestos identification by PLM and dispersion staining in commercial materials

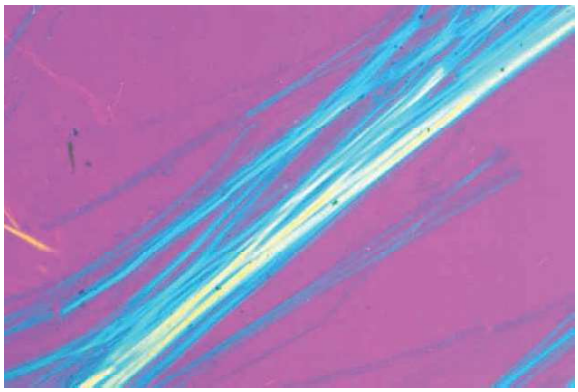


Figure D.1 — PLM micrograph of SRM 1866 chrysotile in 1,550 RI liquid — Crossed polars with 530 nm retardation plate

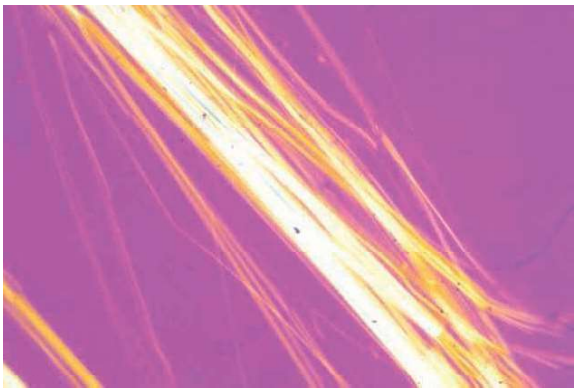


Figure D.2 — PLM micrograph of SRM 1866 chrysotile in 1,550 RI liquid — Crossed polars with 530 nm retardation plate

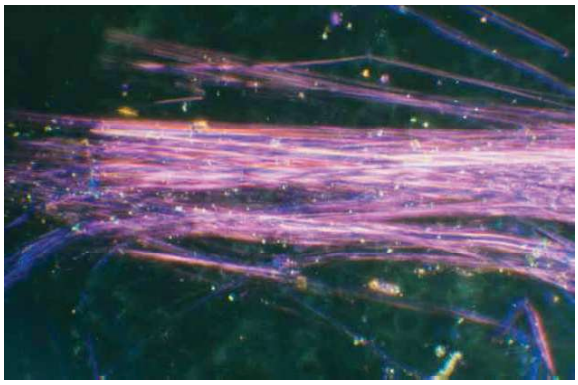


Figure D.3 — SRM 1866 chrysotile in 1,550 RI liquid viewed in dispersion staining — Fibre length parallel to polarizer vibration direction

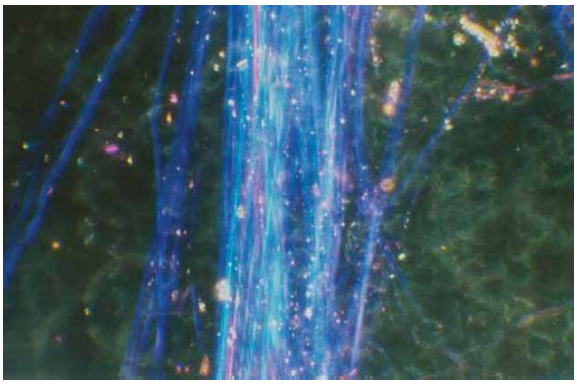


Figure D.4 — SRM 1866 chrysotile in 1,550 RI liquid viewed in dispersion staining — Fibre length normal to polarizer vibration direction

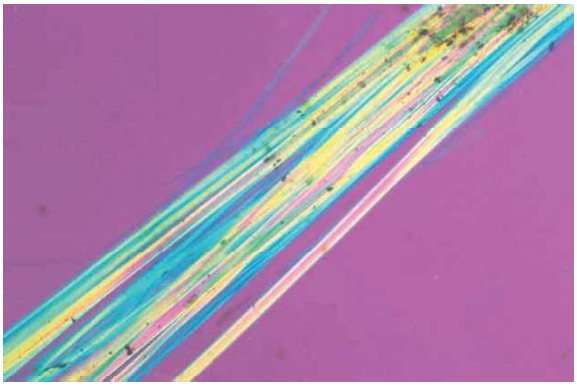


Figure D.5 — PLM micrograph of SRM 1866 amosite in 1,680 RI liquid — Crossed polars with 530 nm retardation plate

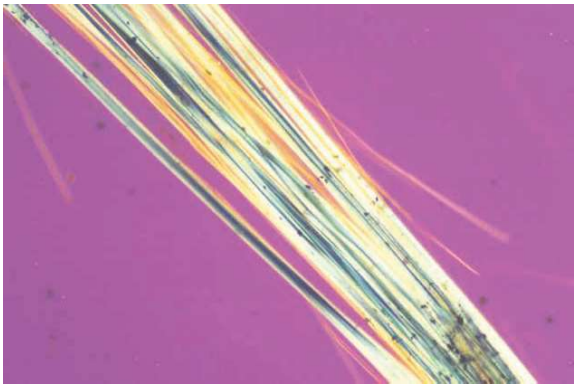


Figure D.6 — PLM micrograph of SRM 1866 amosite in 1,680 RI liquid — Crossed polars with 530 nm retardation plate

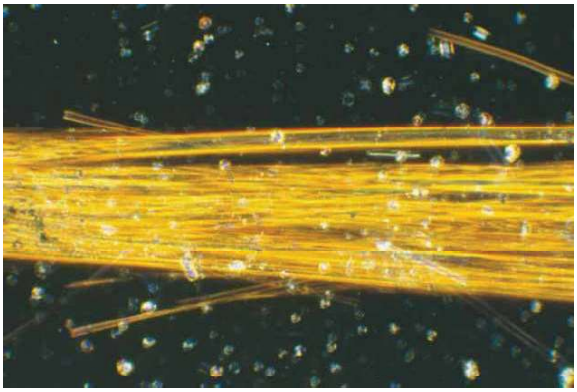


Figure D.7 — SRM 1866 amosite in 1,680 RI liquid viewed in dispersion staining — Fibre length parallel to polarizer vibration direction

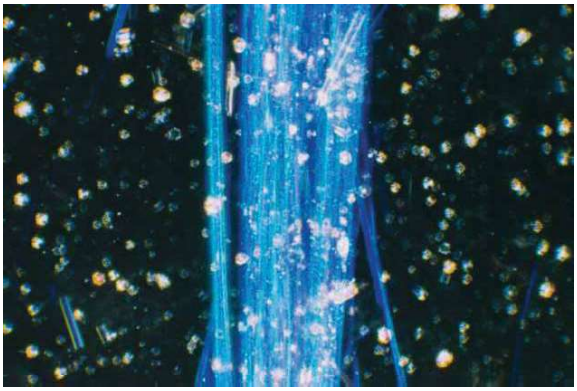


Figure D.8 — SRM 1866 amosite in 1,680 RI liquid viewed in dispersion staining — Fibre length normal to polarizer vibration direction

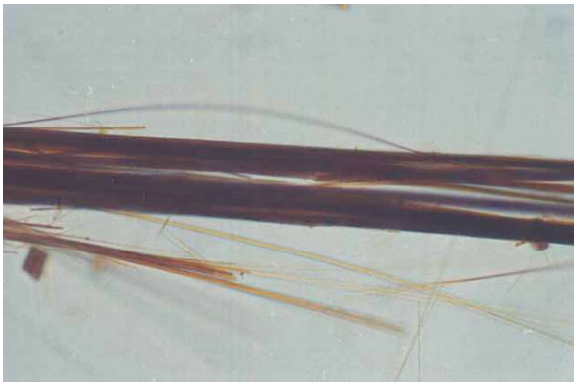


Figure D.9 — Heated amosite in 1,680 RI liquid viewed in plane polarized light — Fibre length parallel to polarizer vibration direction

Figure D.10 — Heated amosite in 1,680 RI liquid viewed in plane polarized light — Fibre length parallel to polarizer vibration direction

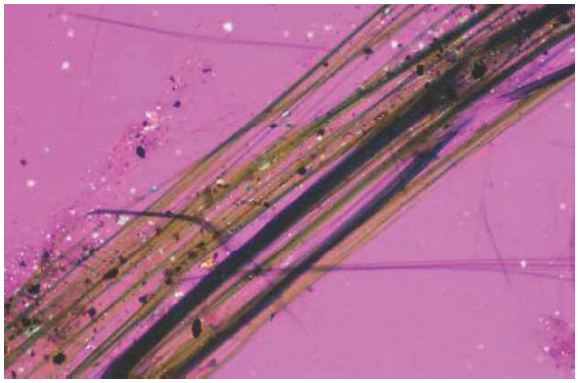


Figure D.11 — PLM micrograph of SRM 1866 crocidolite in 1,700 RI liquid — Crossed polars with 530 nm retardation plate

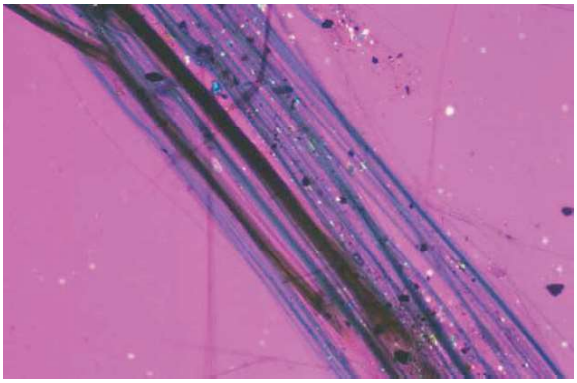


Figure D.12 — PLM micrograph of SRM 1866 crocidolite in 1,700 RI liquid — Crossed polars with 530 nm retardation plate

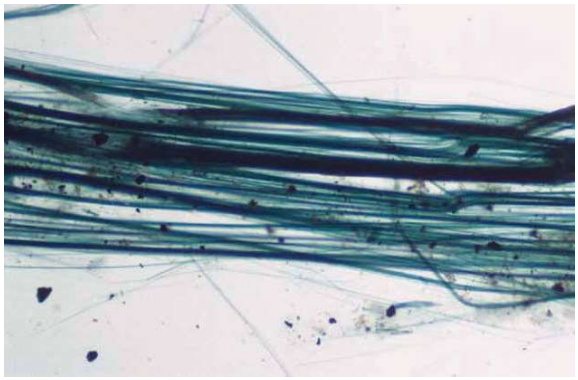


Figure D.13 — SRM 1866 crocidolite in 1,700 RI liquid in plane polarized light — Fibres parallel to polarizer vibration direction

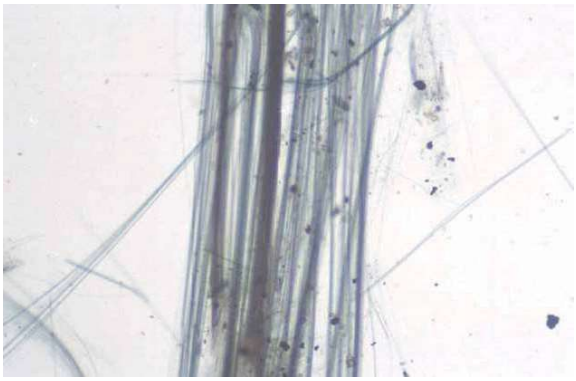


Figure D.14 — SRM 1866 crocidolite in 1,700 RI liquid in plane polarized light — Fibres normal to polarizer vibration direction

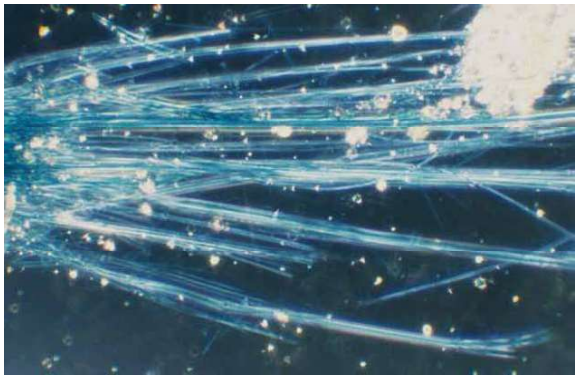


Figure D.15 — SRM 1866 crocidolite in 1,700 RI liquid — Dispersion staining — Fibre lengths parallel to polarizer vibration direction

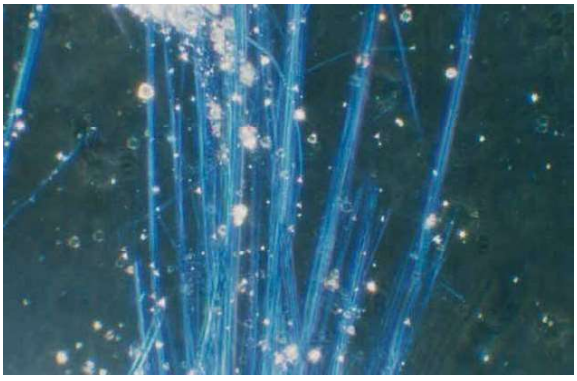


Figure D.16 — SRM 1866 crocidolite in 1,700 RI liquid — Dispersion staining — Fibre lengths normal to polarizer vibration direction

Figure D.17 — Heated crocidolite viewed in plane polarized light — Fibre length parallel to polarizer vibration direction

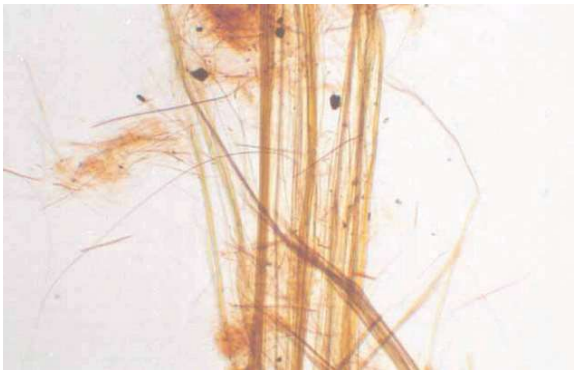


Figure D.18 — Heated crocidolite viewed in plane polarized light — Fibre length normal to polarizer vibration direction

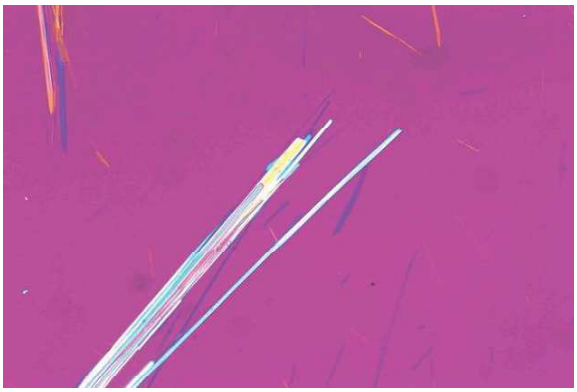


Figure D.19 — PLM micrograph of SRM 1867 tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

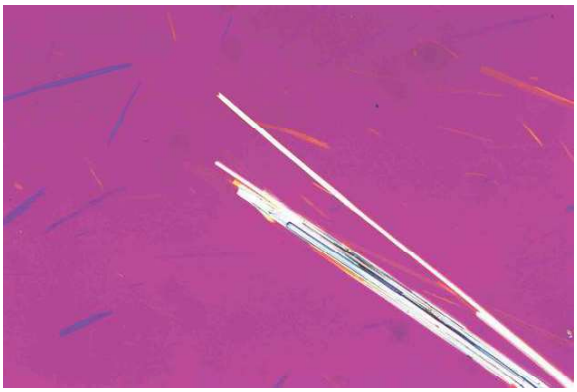


Figure D.20 — PLM micrograph of SRM 1867 tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

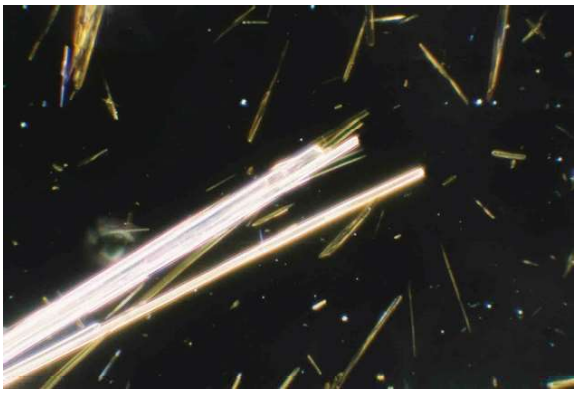


Figure D.21 — SRM 1867 tremolite in 1,605 RI liquid viewed in dispersion staining — Fibres at extinction position

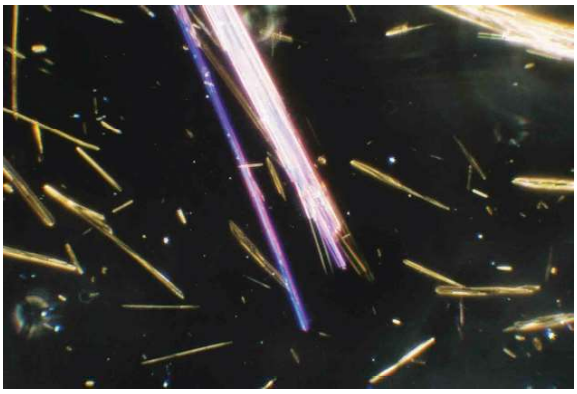


Figure D.22 — SRM 1867 tremolite in 1,605 RI liquid viewed in dispersion staining — Fibres at extinction position

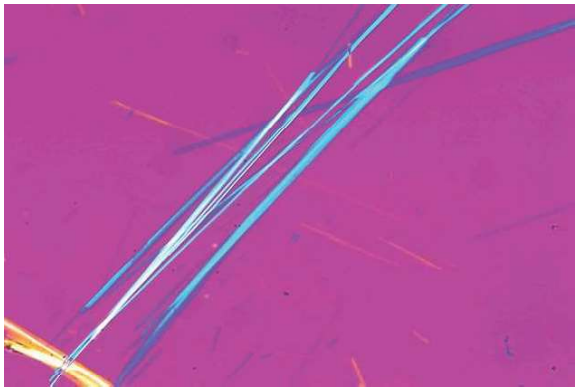


Figure D.23 — PLM micrograph of SRM 1867 tremolite in 1,625 RI liquid — Crossed polars with 530 nm retardation plate

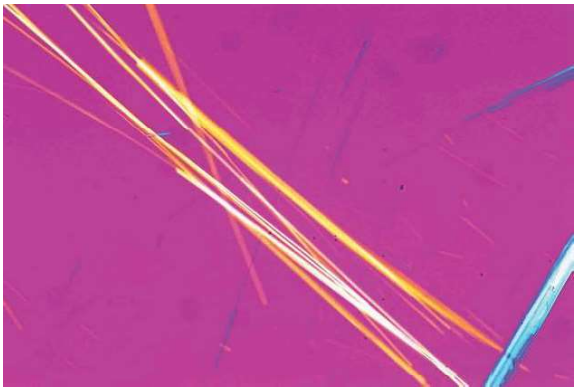


Figure D.24 — PLM micrograph of SRM 1867 tremolite in 1,625 RI liquid — Crossed polars with 530 nm retardation plate

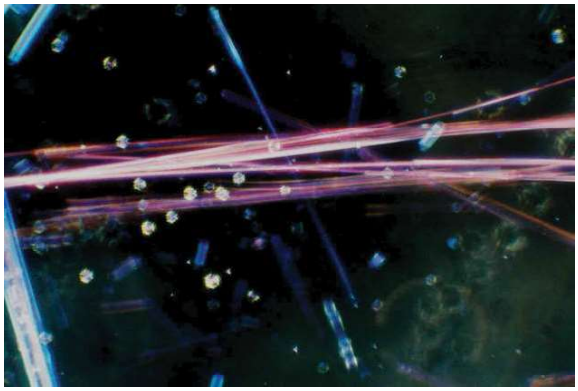


Figure D.25 — SRM 1867 tremolite in 1,625 RI liquid viewed in dispersion staining — Fibres at extinction position

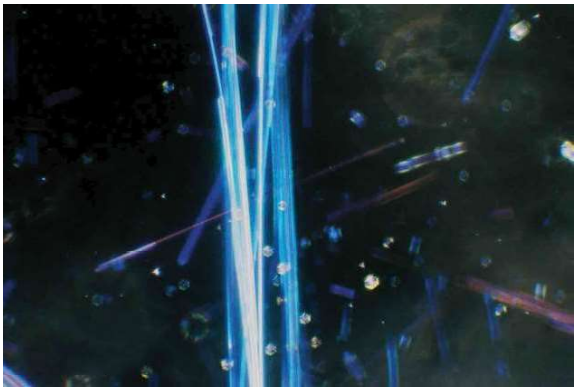


Figure D.26 — SRM 1867 tremolite in 1,625 RI liquid viewed in dispersion staining — Fibres at extinction position

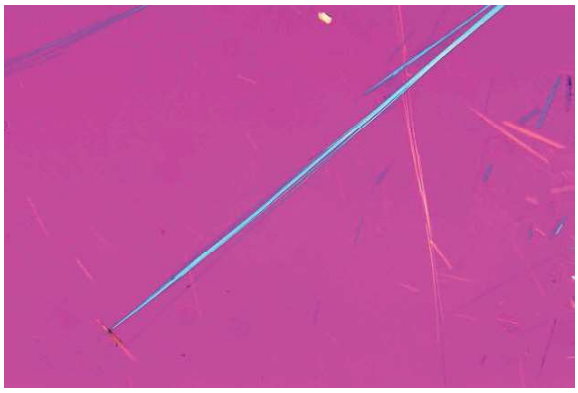


Figure D.27 — PLM micrograph of SRM 1867 actinolite in 1,630 RI liquid — Crossed polars with 530 nm retardation plate

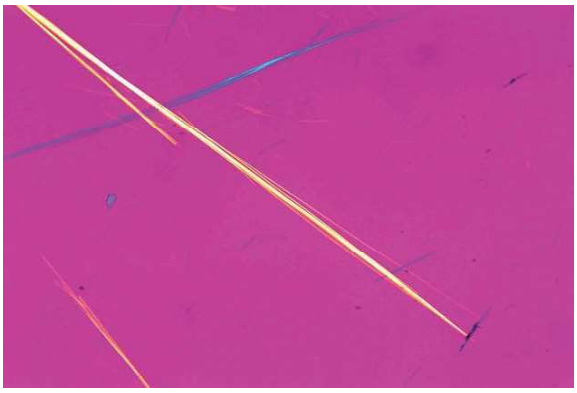


Figure D.28 — PLM micrograph of SRM 1867 actinolite in 1,630 RI liquid — Crossed polars with 530 nm retardation plate

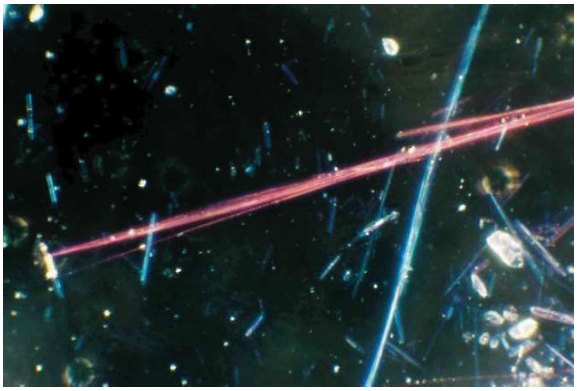


Figure D.29 — SRM 1867 actinolite in 1,630 RI liquid viewed in dispersion staining — Purple fibre at extinction position

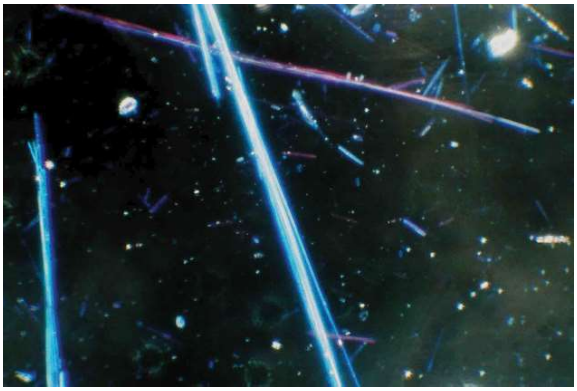


Figure D.30 — SRM 1867 actinolite in 1,630 RI liquid viewed in dispersion staining — Light blue fibre at extinction position

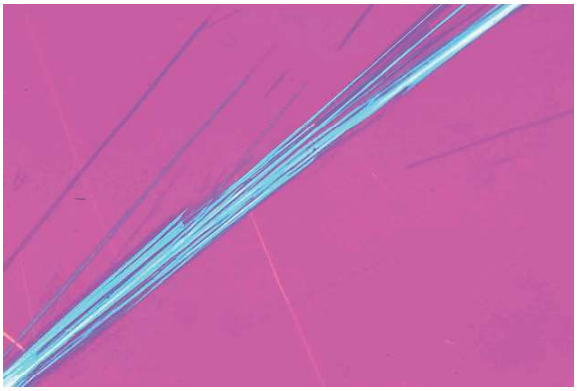


Figure D.31 — PLM micrograph of SRM 1867 anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

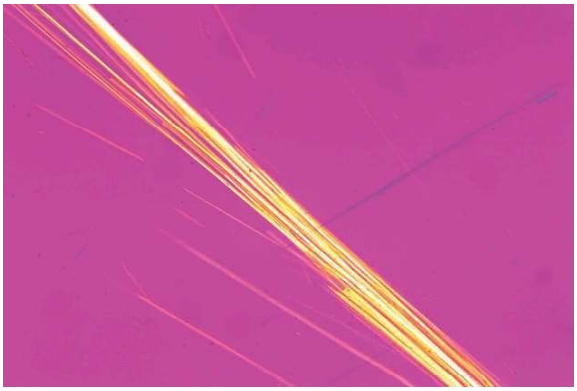


Figure D.32 — PLM micrograph of SRM 1867 anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

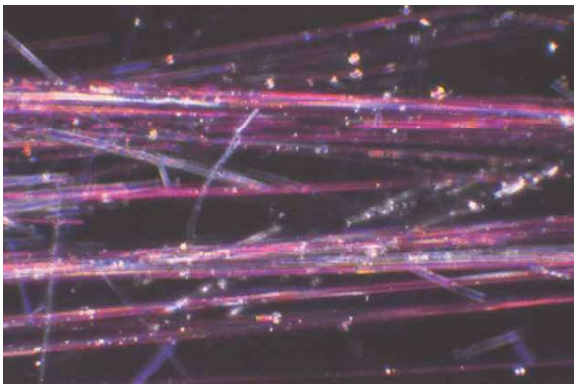


Figure D.33 — SRM 1867 anthophyllite in 1,630 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction

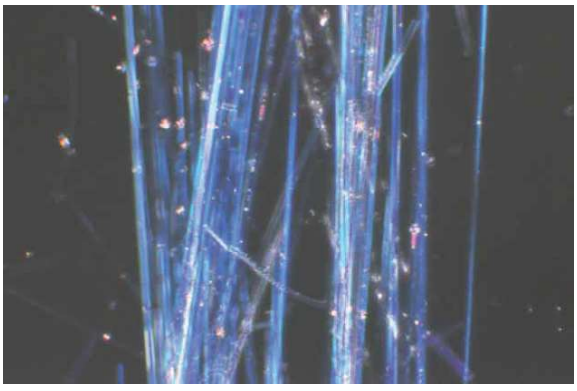


Figure D.34 — SRM 1867 anthophyllite in 1,630 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction

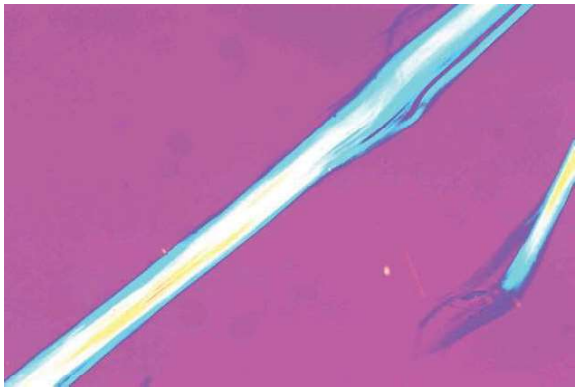


Figure D.35 — PLM micrograph of HSE tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

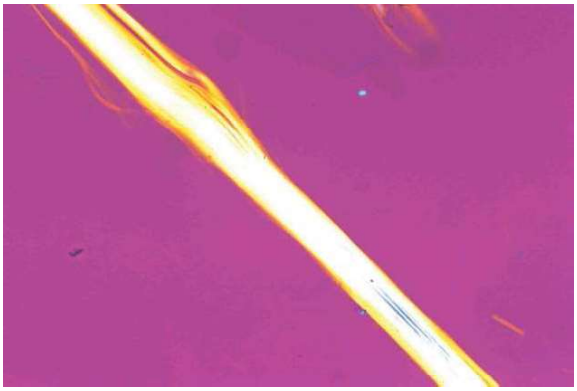


Figure D.36 — PLM micrograph of HSE tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

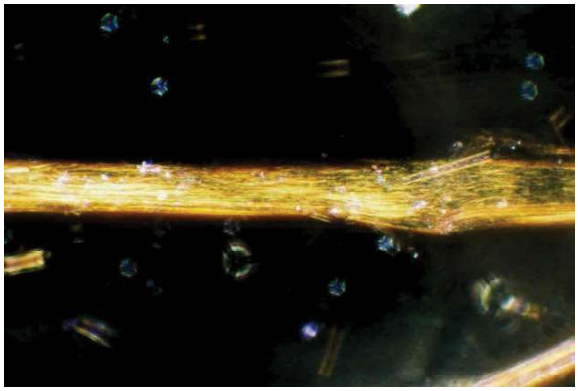


Figure D.37 — HSE tremolite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction

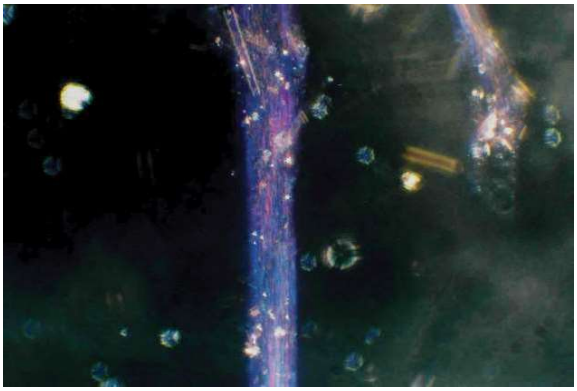


Figure D.38 — HSE tremolite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction

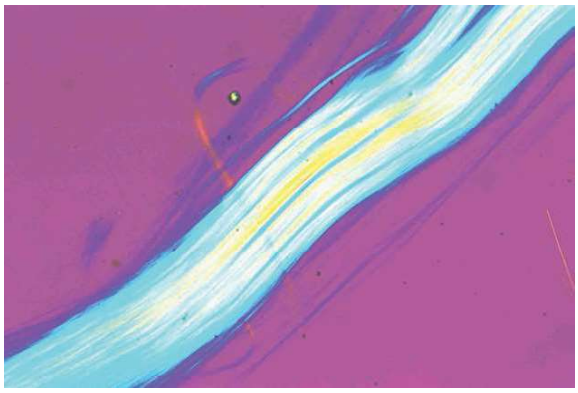


Figure D.39 — PLM micrograph of HSE actinolite in 1,640 RI liquid — Crossed polars with 530 nm retardation plate

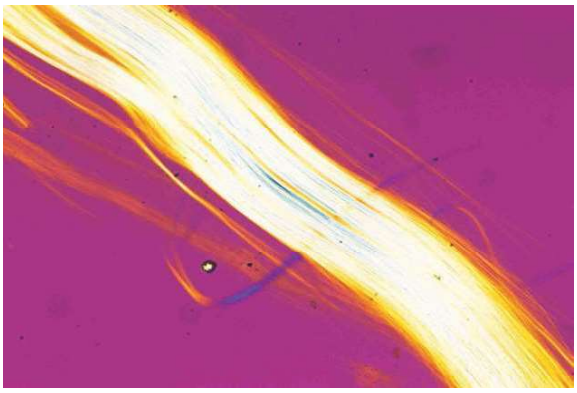


Figure D.40 — PLM micrograph of HSE actinolite in 1,640 RI liquid — Crossed polars with 530 nm retardation plate

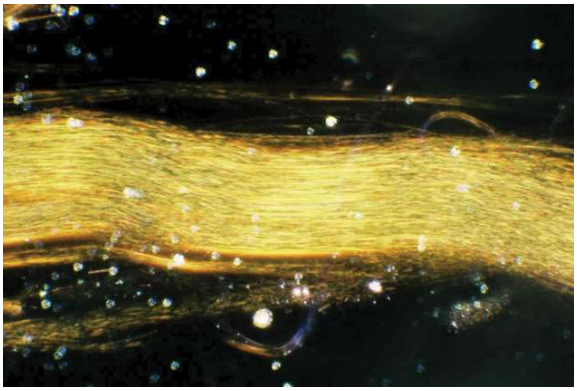


Figure D.41 — HSE actinolite in 1,640 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction

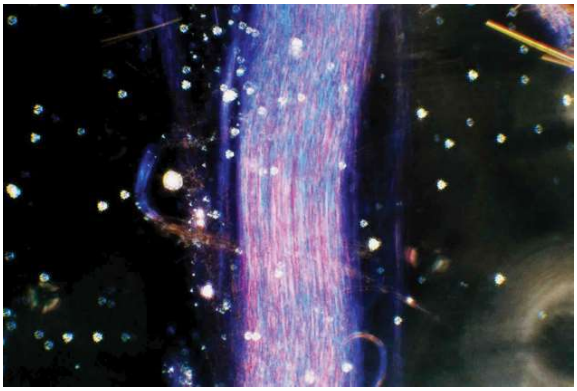


Figure D.42 — HSE actinolite in 1,640 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction

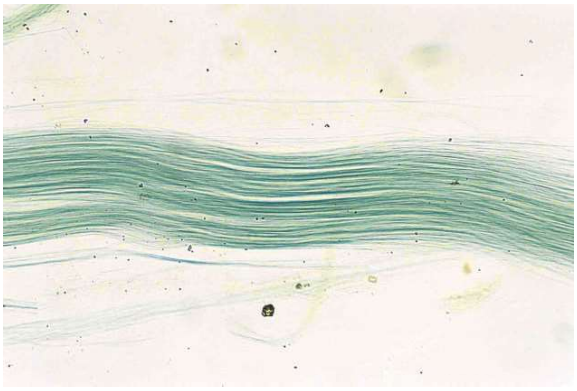


Figure D.43 — HSE actinolite in 1,640 RI liquid in plane polarized light — Fibres parallel to polarizer vibration direction

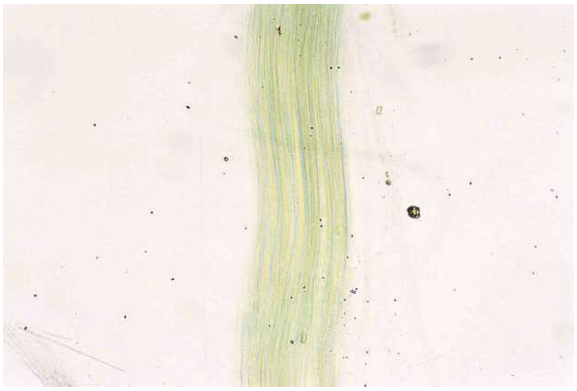


Figure D.44 — HSE actinolite in 1,640 RI liquid in plane polarized light — Fibres normal to polarizer vibration direction

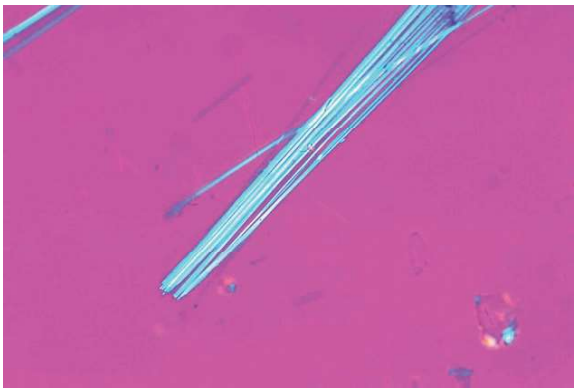


Figure D.45 — PLM micrograph of HSE anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

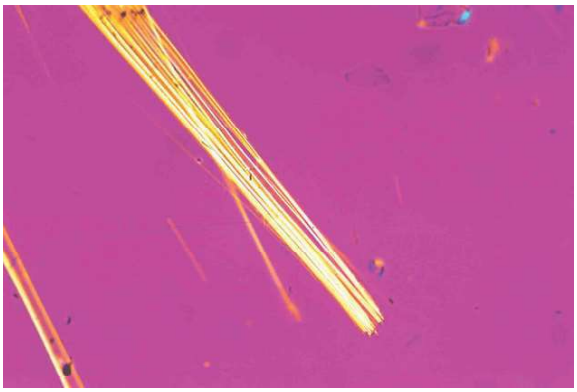


Figure D.46 — PLM micrograph of HSE anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate

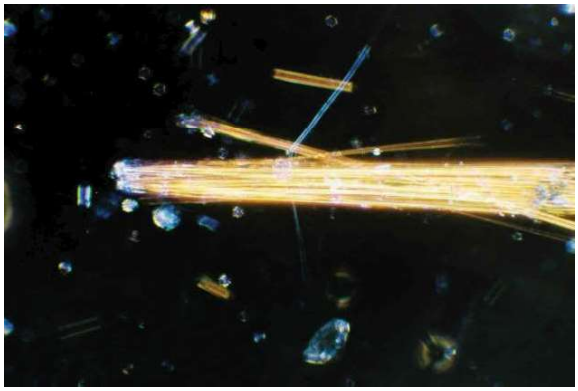


Figure D.47 — HSE anthophyllite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction

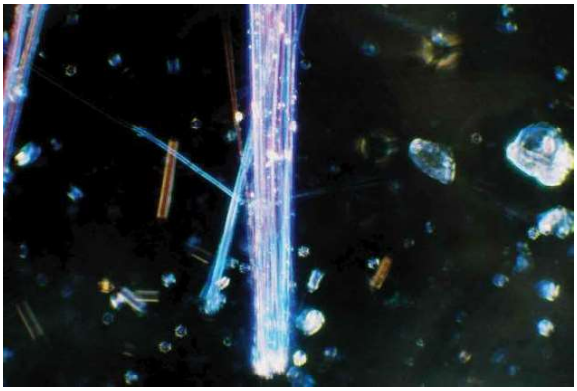


Figure D.48 — HSE anthophyllite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction

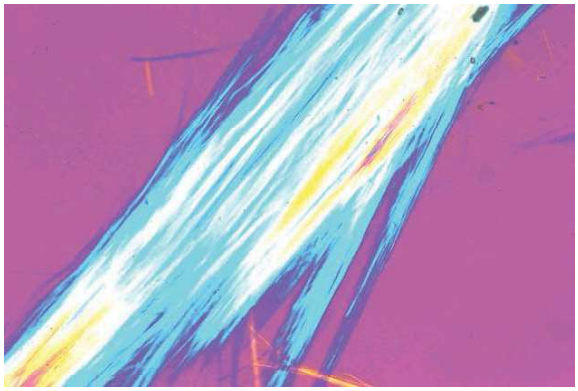


Figure D.49 — PLM micrograph of richterite/winchite asbestos in 1,630 RI liquid — Crossed polars with 530 nm retardation plate

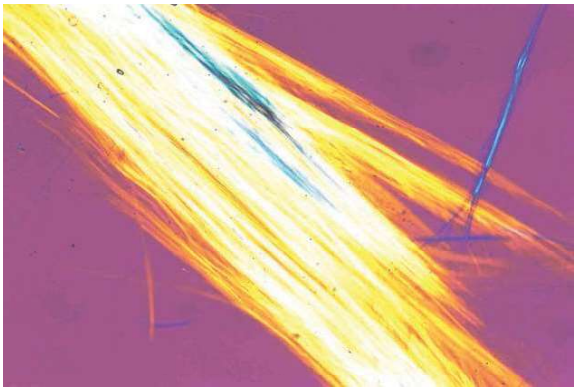


Figure D.50 — PLM micrograph of richterite/winchite asbestos in 1,630 RI liquid — Crossed polars with 530 nm retardation plate

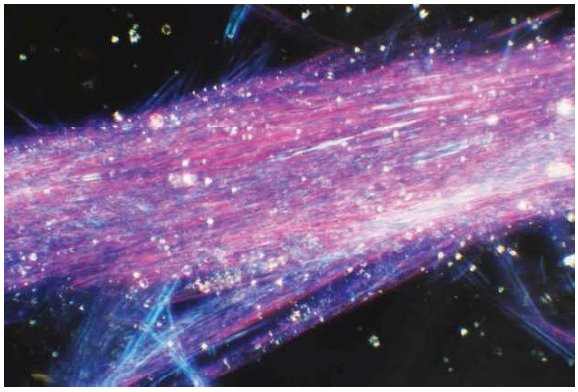


Figure D.51 — Richterite/winchite asbestos in 1,630 RI liquid viewed in dispersion staining — Fibres at extinction position

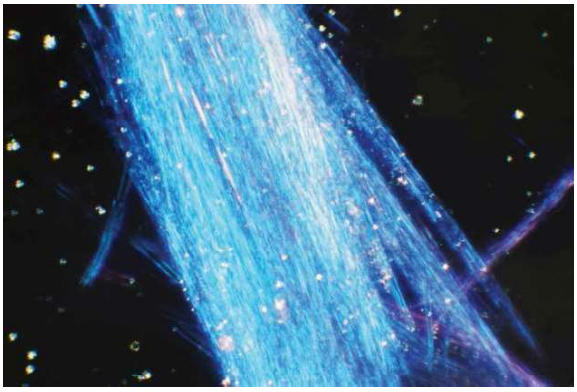


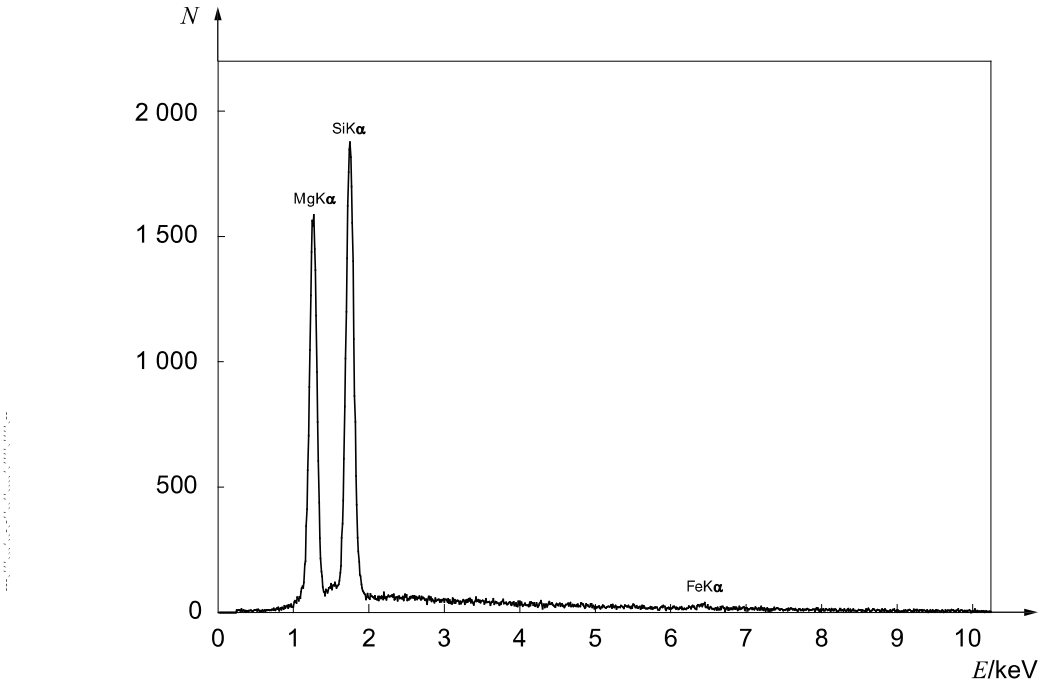
Figure D.52 — Richterite/winchite asbestos in 1,630 RI liquid viewed in dispersion staining — Fibres at extinction position

Annex E
(normative)

Asbestos identification by SEM in commercial materials

Figures E.1 to E.11 are examples of EDXA spectra collected on an SEM operating at 15 kV and using a silicon solid-state detector with a beryllium window. The SEM specimens were prepared by mounting representative fibre bundles from SRM 1866, SRM 1867, and the HSE reference asbestos varieties on adhesive tape on SEM specimen stubs. All specimens were carbon coated in a vacuum evaporator.

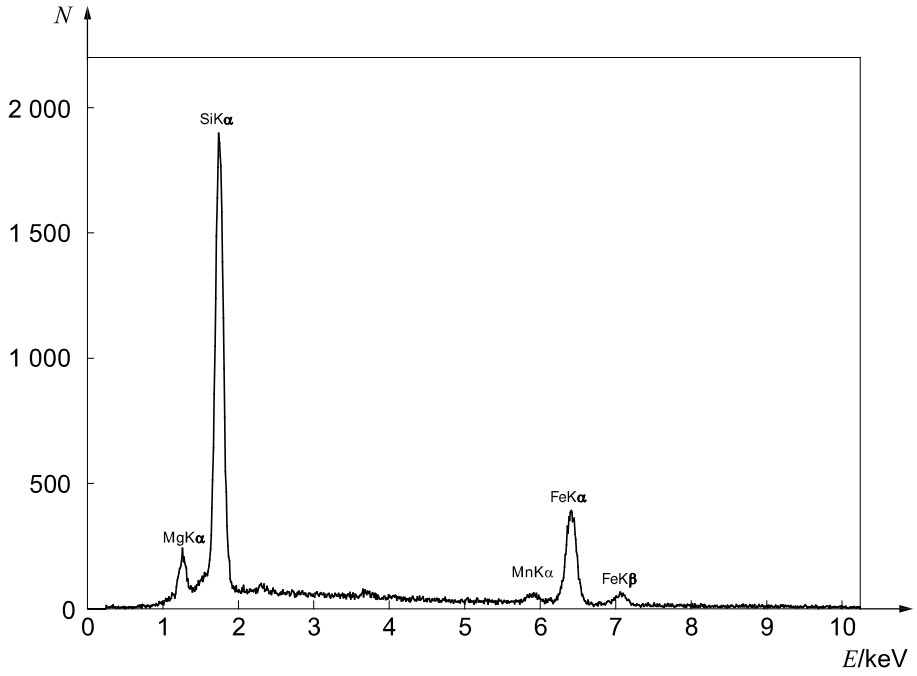
Prior to use of this part of ISO 22262, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific X-ray detector.



Key

N counts E X-ray energy

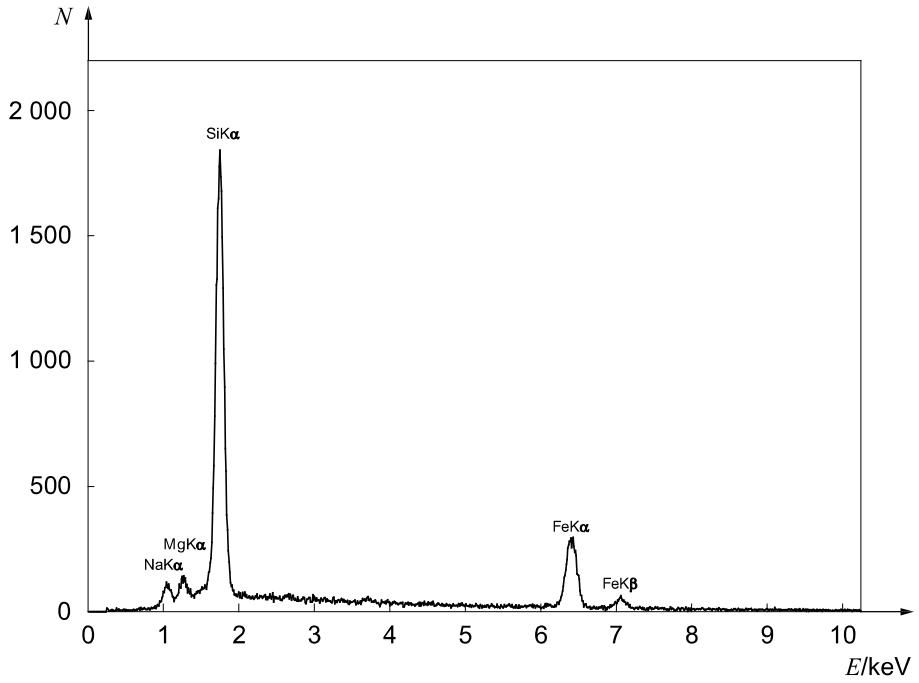
Figure E.1 — Energy dispersive X-ray spectrum obtained from SRM 1866 chrysotile



Key

N counts E X-ray energy

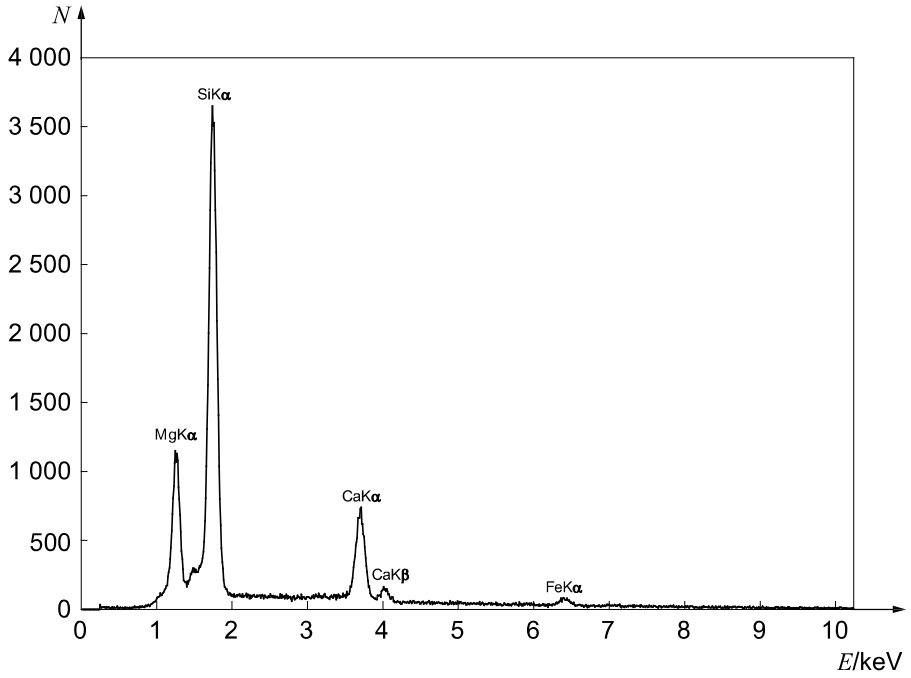
Figure E.2 — Energy dispersive X-ray spectrum obtained from SRM 1866 amosite



Key

N counts E X-ray energy

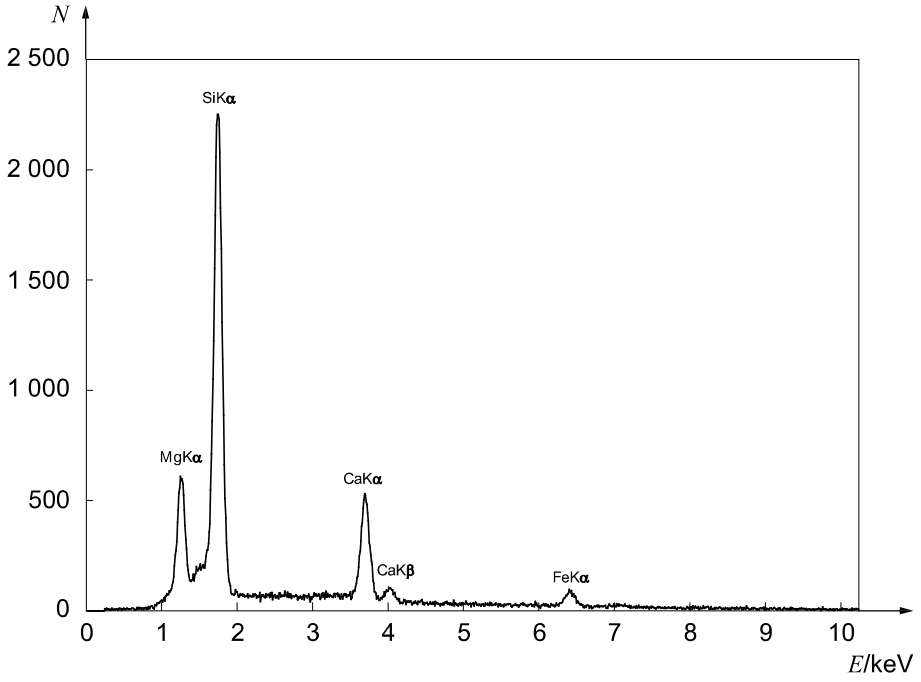
Figure E.3 — Energy dispersive X-ray spectrum obtained from SRM 1866 crocidolite



Key

N counts E X-ray energy

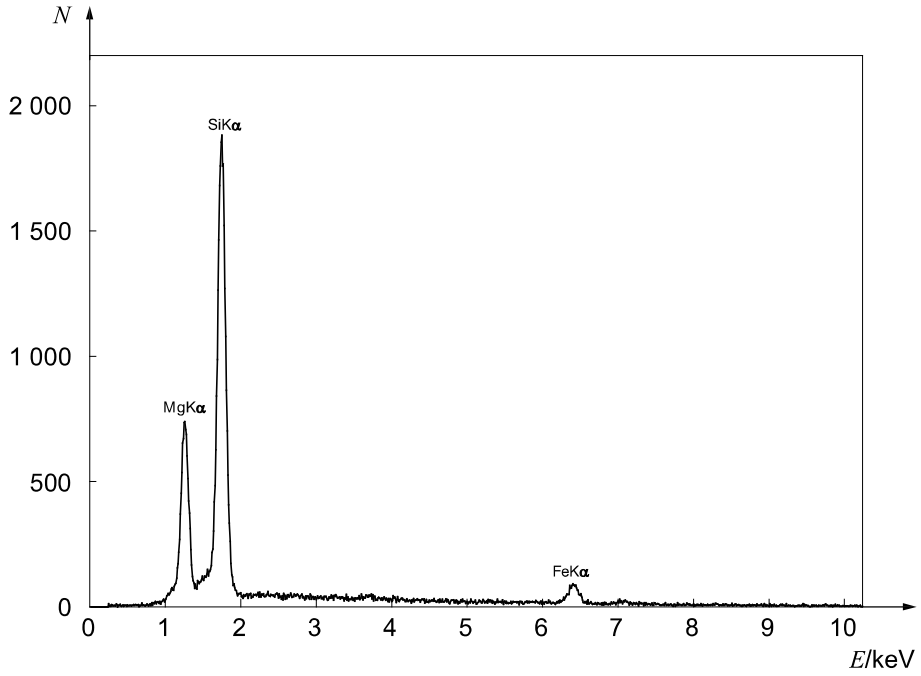
Figure E.4 — Energy dispersive X-ray spectrum obtained from SRM 1867 tremolite



Key

N counts E X-ray energy

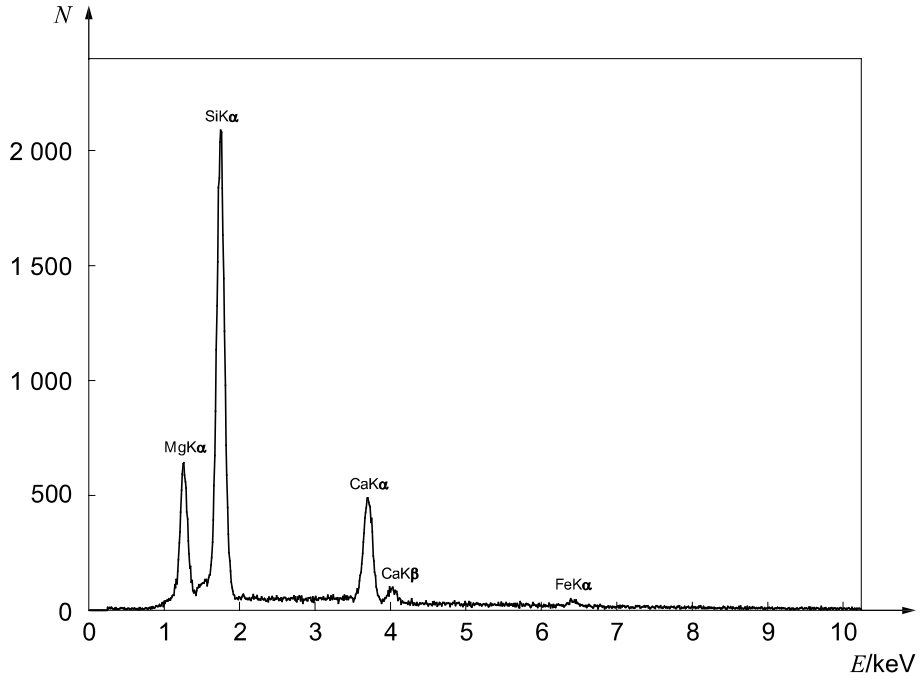
Figure E.5 — Energy dispersive X-ray spectrum obtained from SRM 1867 actinolite



Key

N counts E X-ray energy

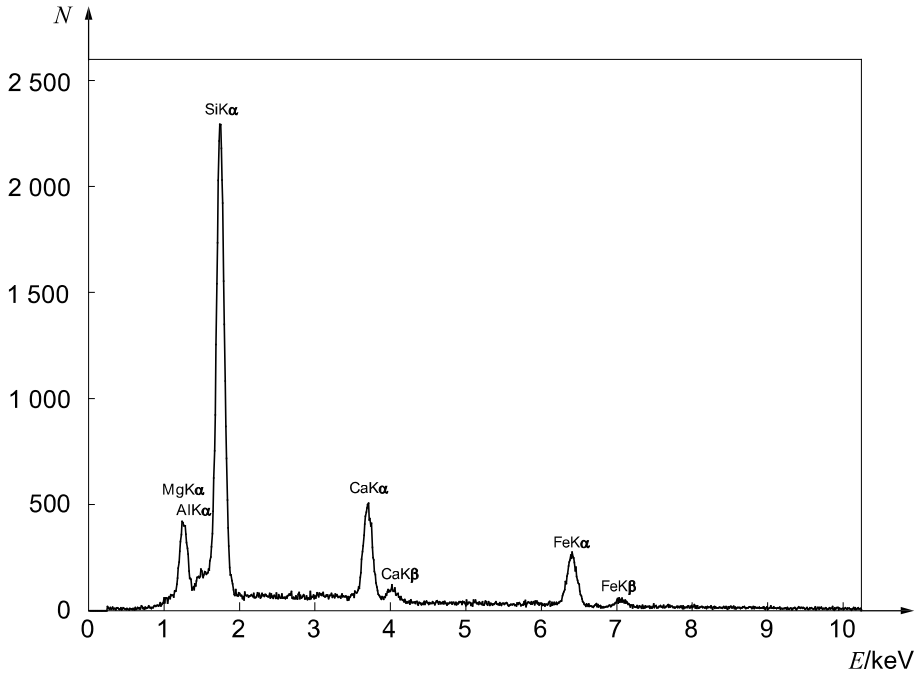
Figure E.6 — Energy dispersive X-ray spectrum obtained from SRM 1867 anthophyllite



Key

N counts E X-ray energy

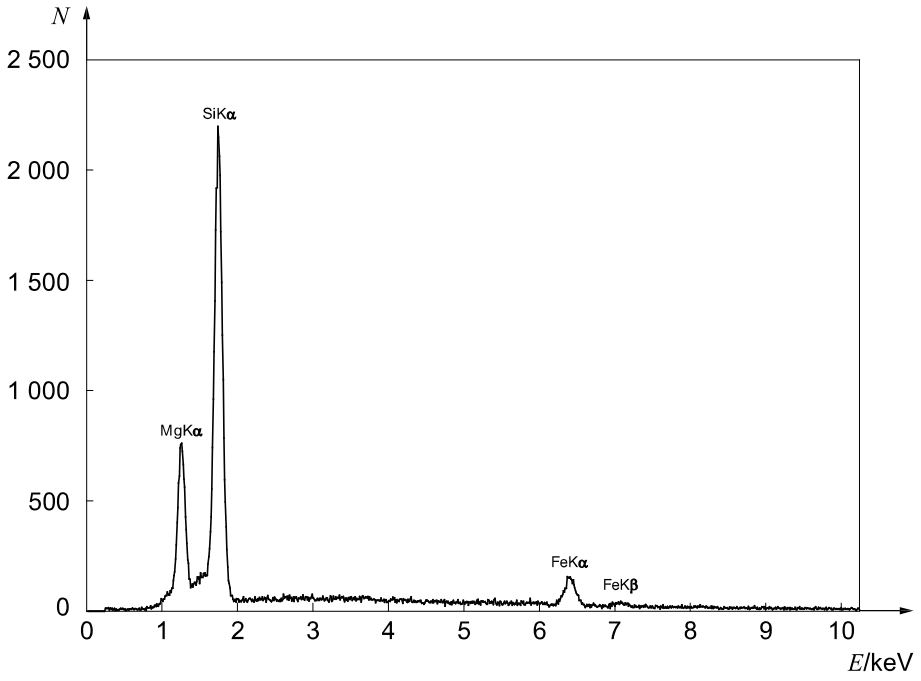
Figure E.7 — Energy dispersive X-ray spectrum obtained from HSE tremolite



Key

N counts E X-ray energy

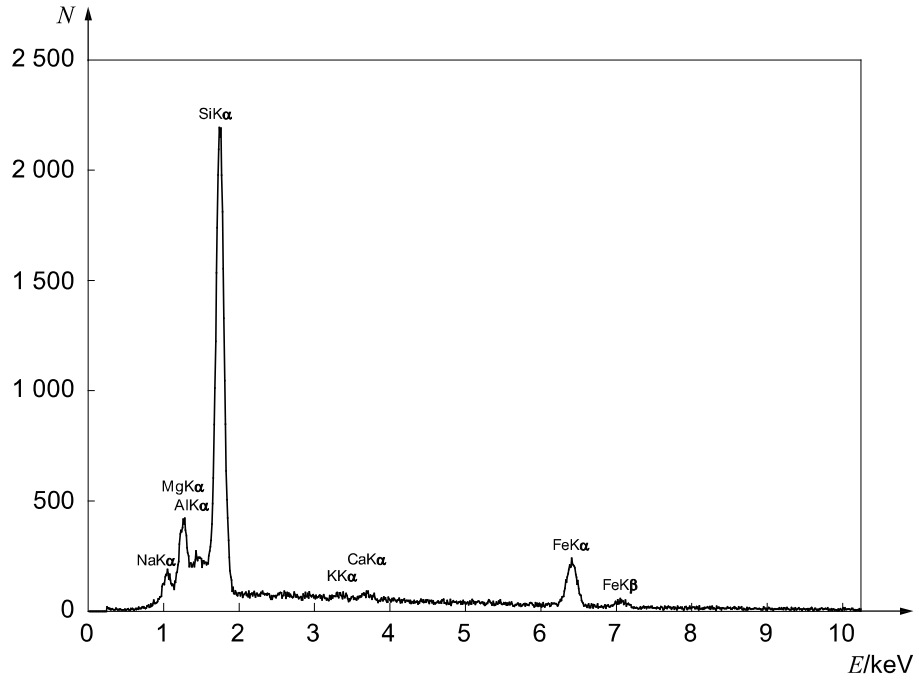
Figure E.8 — Energy dispersive spectrum obtained from HSE actinolite



Key

N counts E X-ray energy

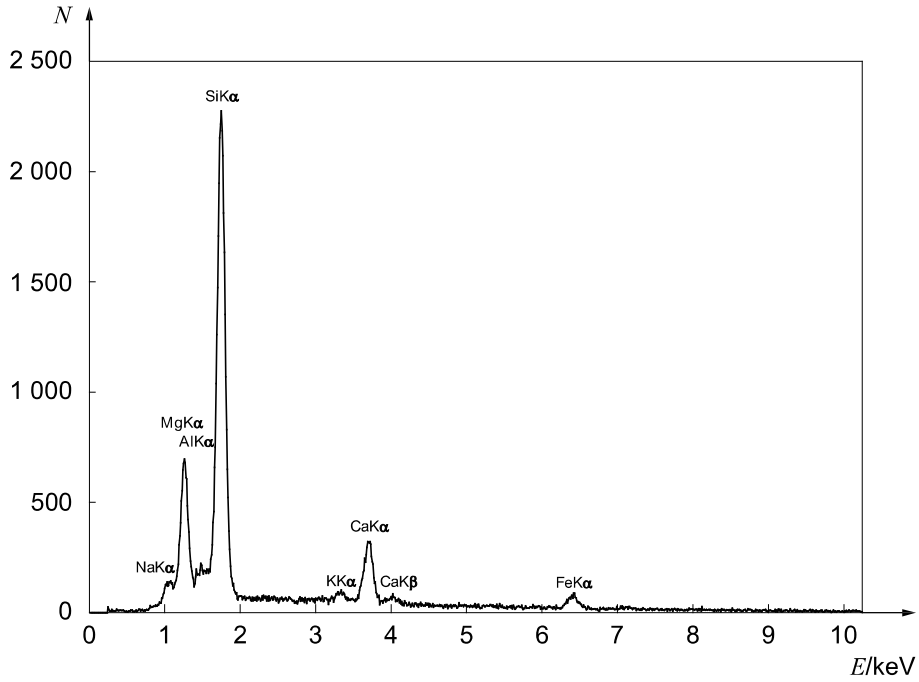
Figure E.9 — Energy dispersive X-ray spectrum obtained from HSE anthophyllite



Key

N counts E X-ray energy

Figure E.10 — Energy dispersive X-ray spectrum obtained from Bolivian crocidolite



Key

N counts E X-ray energy

Figure E.11 — Energy dispersive X-ray spectrum obtained from richterite/winchite

Annex F
(normative)

Asbestos identification by TEM in commercial materials

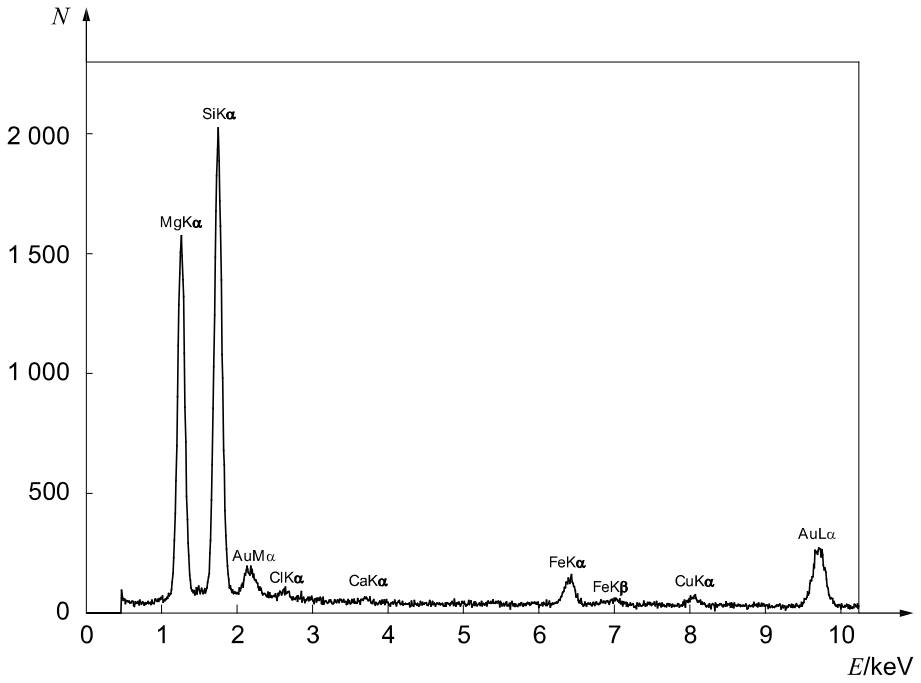
F.1 General

For the identification of asbestos in some types of bulk materials, particularly for those in which PLM examination yields ambiguous results, TEM examination can usually resolve the ambiguities and provide definitive identification of the fibres. In most cases, acquisition of an EDXA spectrum provides sufficient evidence to identify any of the asbestos varieties. Discrimination between talc and anthophyllite, however, cannot be reliably achieved on the basis of an EDXA spectrum alone, because the chemical compositions of the two minerals are very similar. Electron diffraction permits discrimination between talc and anthophyllite on the basis of their different crystal structures.

F.2 EDXA analysis

Figures F.1 to F.11 are examples of EDXA spectra collected on a TEM operating at 80 kV and using a silicon solid state detector with a beryllium window. The TEM specimens were prepared by the micropipette method from SRM 1866, SRM 1867 and HSE reference asbestos varieties. All specimens were prepared using gold grids in order to avoid interference in detection of the Na K_α peak by the Cu L_α peak which would partially overlap the sodium peak if copper specimen grids were used.

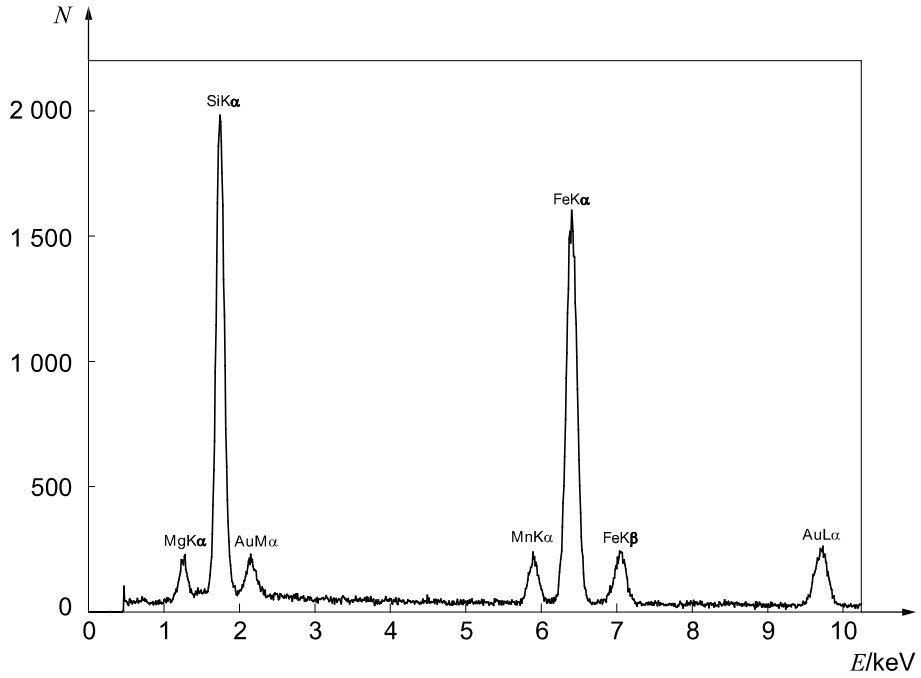
Prior to use of this part of ISO 22262, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific X-ray detector.



Key

N counts E X-ray energy

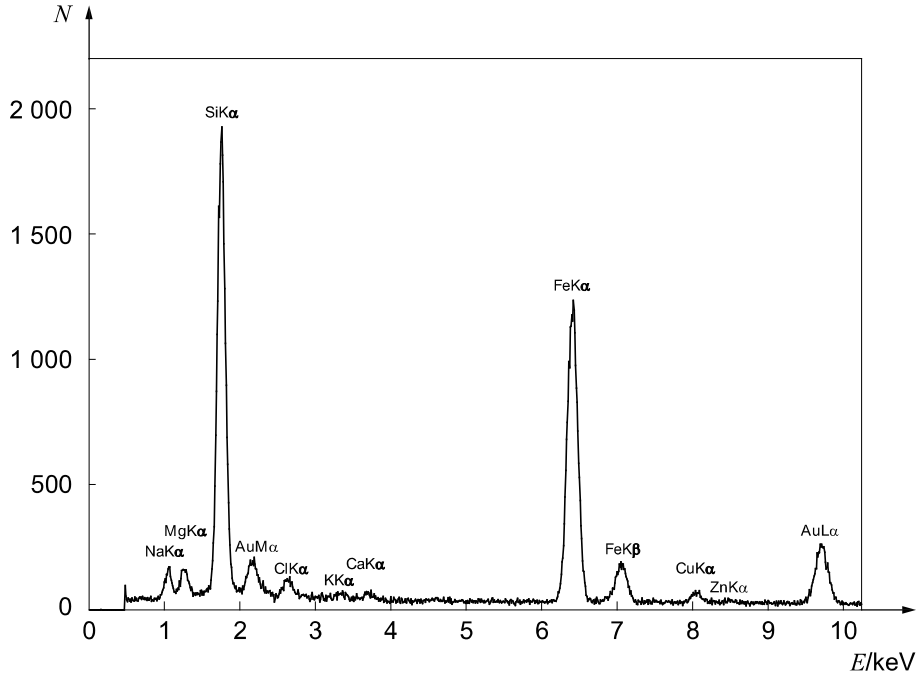
Figure F.1 — Energy dispersive X-ray spectrum obtained from SRM 1866 chrysotile. The gold and small copper peaks originate from the gold specimen grid



Key

N counts E X-ray energy

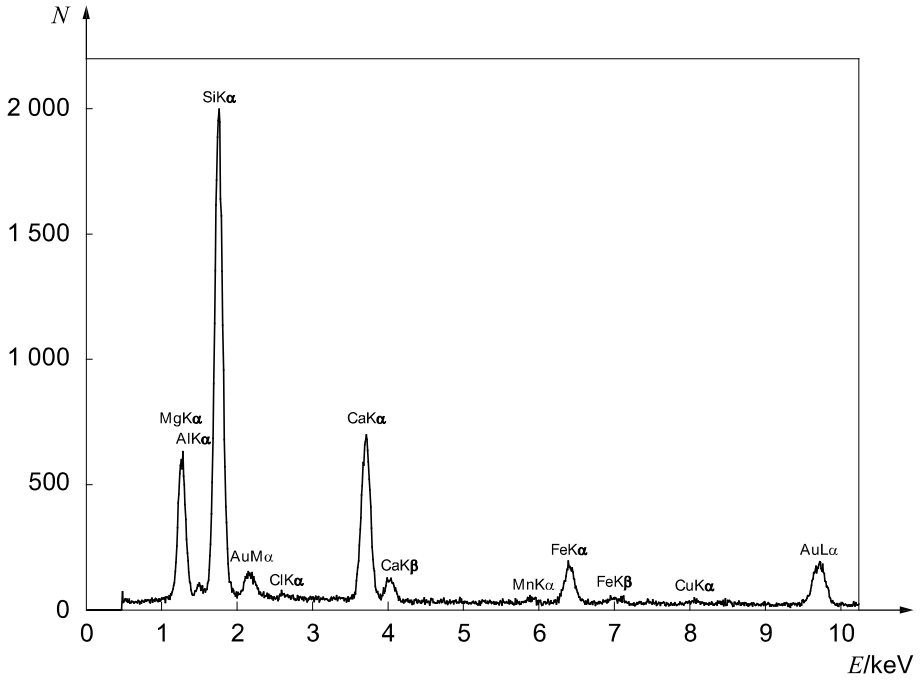
Figure F.2 — Energy dispersive X-ray spectrum obtained from SRM 1866 amosite. The gold peaks originate from the gold specimen grid



Key

N counts E X-ray energy

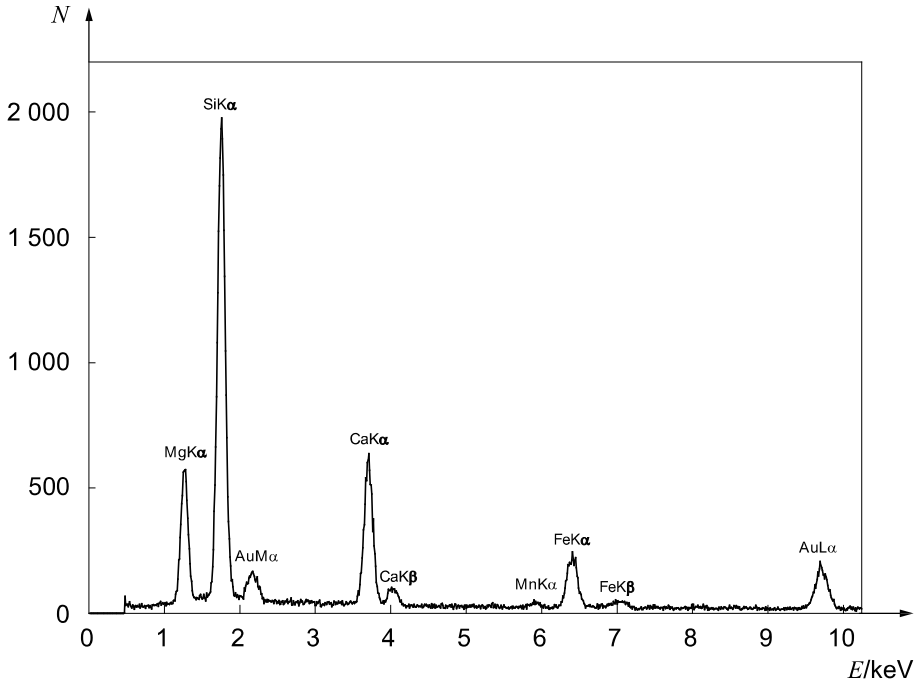
Figure F.3 — Energy dispersive X-ray spectrum obtained from SRM 1866 crocidolite. The gold and small copper peaks originate from the gold specimen grid



Key

N counts E X-ray energy

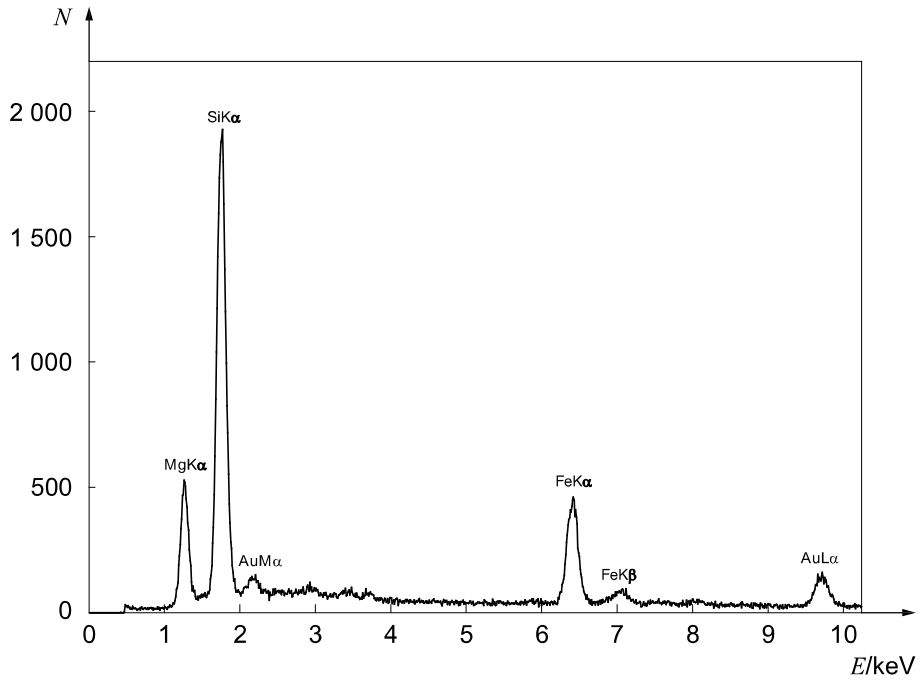
Figure F.4 — Energy dispersive X-ray spectrum obtained from SRM 1867 tremolite. The gold and small copper peaks originate from the gold specimen grid



Key

N counts E X-ray energy

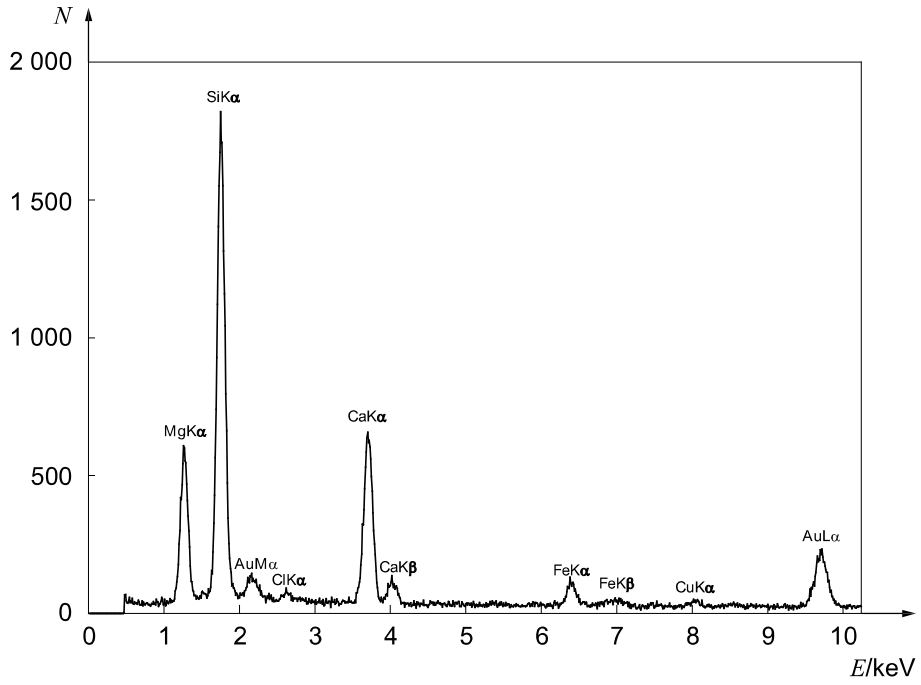
Figure F.5 — Energy dispersive X-ray spectrum obtained from SRM 1867 actinolite. The gold peaks originate from the gold specimen grid



Key

N counts E X-ray energy

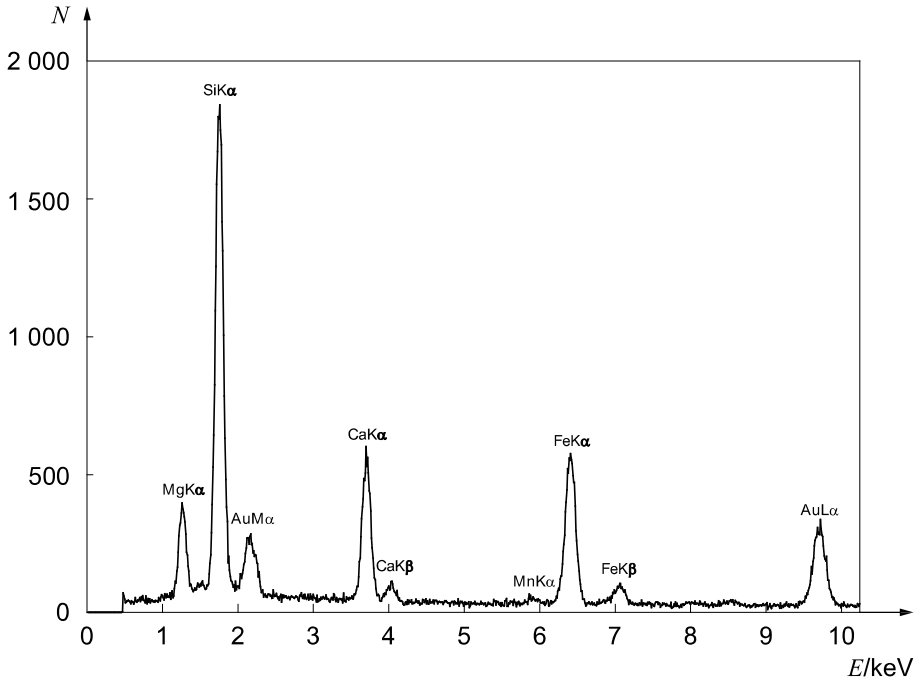
Figure F.6 — Energy dispersive X-ray spectrum obtained from SRM 1867 anthophyllite. The gold peaks originate from the gold specimen grid



Key

N counts E X-ray energy

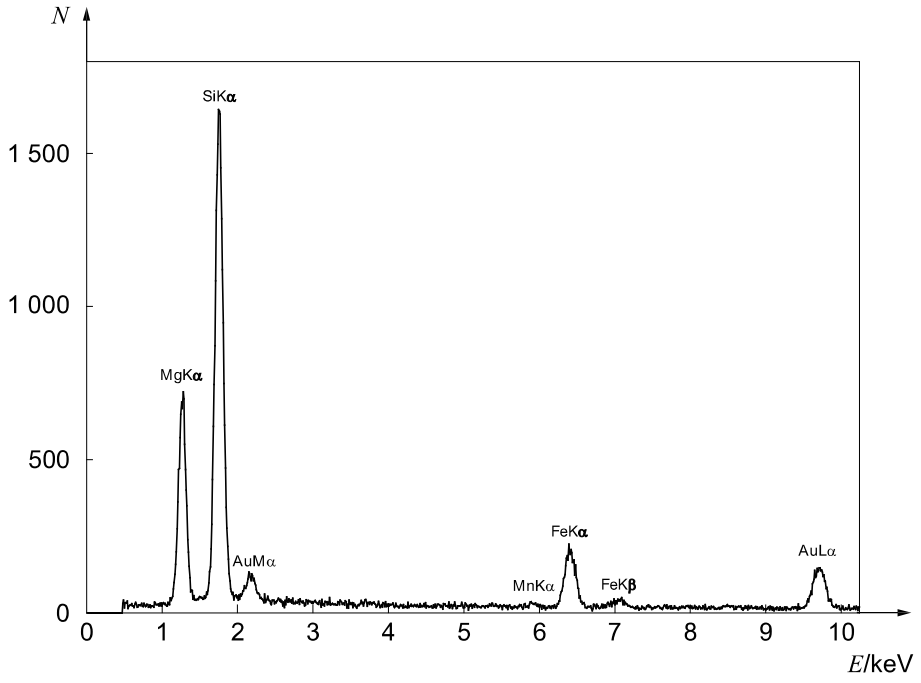
Figure F.7 — Energy dispersive X-ray spectrum obtained from HSE tremolite. The gold and small copper peaks originate from the gold specimen grid



Key

N counts E X-ray energy

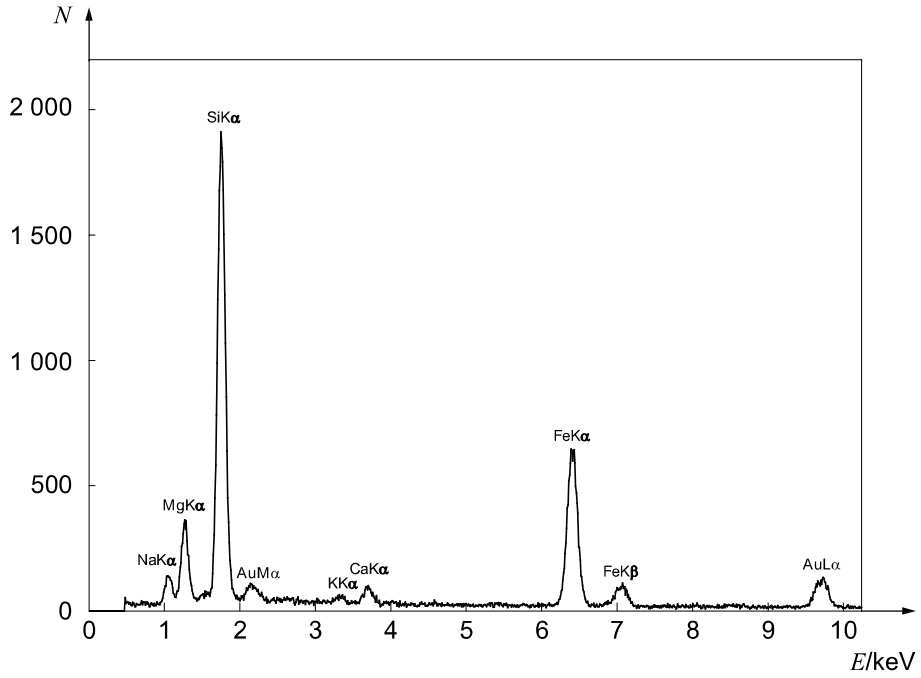
Figure F.8 — Energy dispersive X-ray spectrum obtained from HSE actinolite. The gold peaks originate from the the gold specimen grid



Key

N counts E X-ray energy

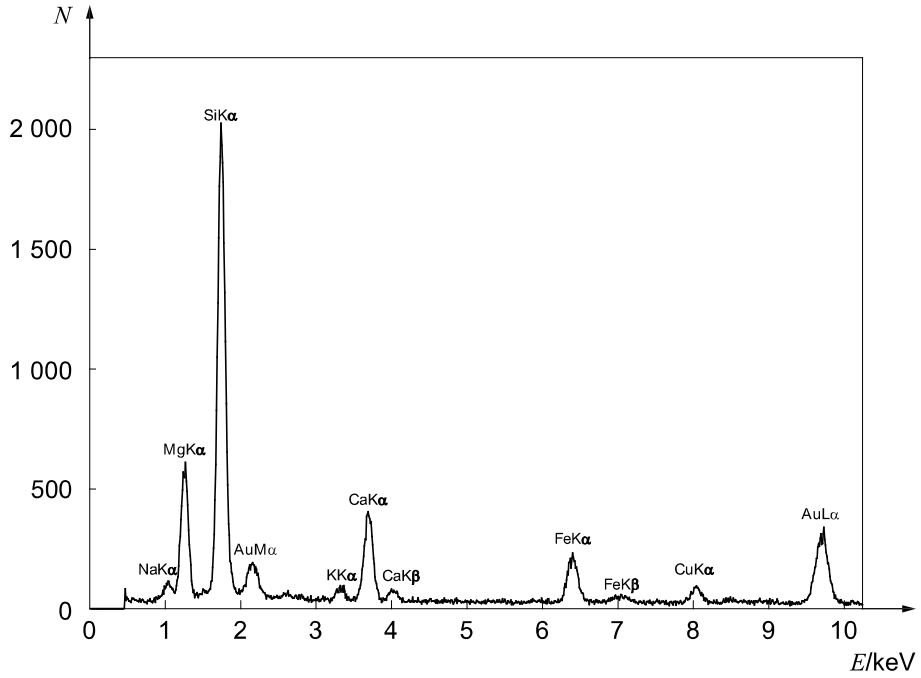
Figure F.9 — Energy dispersive X-ray spectrum obtained from HSE anthophyllite. The gold peaks originate from the gold specimen grid



Key

N counts E X-ray energy

Figure F.10 — Energy dispersive X-ray spectrum obtained from Bolivian crocidolite. The gold peaks originate from the gold specimen grid



Key

N counts E X-ray energy

Figure F.11 — Energy dispersive X-ray spectrum obtained from richterite/winchite asbestos. The gold and small copper peaks originate from the gold specimen grid

F.3 Electron diffraction

The ED technique can be either qualitative or quantitative. Qualitative ED consists of visual examination, without detailed measurement, of the general characteristics of the ED pattern obtained on the TEM viewing screen from a randomly oriented fibre. ED patterns obtained from fibres with cylindrical symmetry, such as chrysotile, do not change when the fibres are tilted about their axes, and patterns from randomly oriented fibres of these minerals can be interpreted quantitatively. For fibres which do not have cylindrical symmetry, only those ED patterns obtained when the fibre is oriented with a principal crystallographic axis closely parallel to the incident electron-beam direction can be interpreted quantitatively. This type of ED pattern shall be referred to as a zone-axis ED pattern. In order to interpret a zone-axis ED pattern quantitatively, it shall be recorded photographically and its consistency with known mineral structures shall be checked. A computer program may be used to compare measurements of the zone-axis ED pattern with corresponding data calculated from known mineral structures. The zone-axis ED pattern obtained by examination of a fibre in a particular orientation can be insufficiently specific to permit unequivocal identification of the mineral fibre, but it is often possible to tilt the fibre to another angle and to record a different ED pattern corresponding to another zone axis. The angle between the two zone axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the ED pattern, the camera length of the TEM should be set to a low value of approximately 250 mm and the ED pattern should then be viewed through the binoculars. This procedure minimizes the possible degradation of the fibre by the electron irradiation. However, the pattern is distorted by the tilt angle of the viewing screen. A camera length of at least 2 m should be used when the ED pattern is recorded, if accurate measurement of the pattern is to be possible. It is necessary that, when obtaining an ED pattern to be evaluated visually or recorded, the sample height shall be properly adjusted to the eucentric point and the image shall be focused in the plane of the selected area aperture. If this is not done, there may be some components of the ED pattern which do not originate from the selected area. In general, it is necessary to use the smallest available ED aperture.

For accurate measurements of the ED pattern, it is recommended that an internal calibration standard be used. Apply a thin coating of gold, or other suitable calibration material, to the underside of the TEM specimen. This coating may be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every ED pattern and these rings provide the required calibration information. Alternatively, a calibrated objective aperture can be inserted to determine if the layer-line spacing of the ED pattern is approximately 0,53 nm, as expected for asbestos fibres (Reference [30]). This works well even when viewing a raised screen through binoculars.

To form an ED pattern, move the image of the fibre to the centre of the viewing screen, adjust the height of the specimen to the eucentric position, and insert a suitable selected area aperture into the electron beam so that the fibre, or a portion of it, occupies a large proportion of the illuminated area. The size of the aperture and the portion of the fibre shall be such that particles other than the one to be examined are excluded from the selected area. Observe the ED pattern through the binoculars. During the observation, the objective lens current should be adjusted to the point where the most complete ED pattern is obtained. If an incomplete ED pattern is still obtained, move the particle around within the selected area to attempt to optimize the ED pattern, or to eliminate possible interferences from neighbouring particles.

ED patterns can be particularly useful for differentiating fibrous talc from anthophyllite asbestos, both of which have similar EDXA spectra. ED of talc produces a pseudo-hexagonal pattern that does not change as the fibre is tilted using the goniometer. Anthophyllite asbestos, on the other hand, produces assorted spots appearing and disappearing along layer lines as the fibre is tilted using the goniometer. ED patterns can also be a useful diagnostic tool for chrysotile that is so heavily coated with matrix that EDXA is inconclusive. Detection of the 002, 110, and 130 reflections as shown in Figure F.12 in conjunction with 0,53 nm layer-line spacing confirms the presence of chrysotile.

Analysis of laboratory samples seldom requires zone-axis measurements. However, if a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient holder allows complete rotation of the specimen grid and tilting of the grid about a single axis. Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED pattern appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis

conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of twinning or multiple

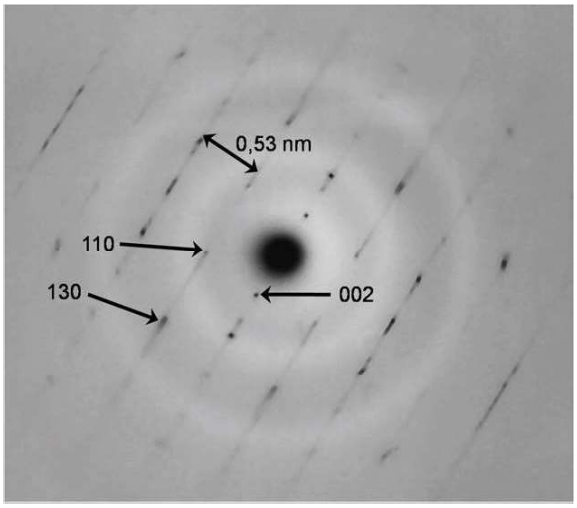


Figure F.12 — Chrysotile SAED pattern

diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. A full discussion of electron diffraction and multiple diffraction can be found in References [26]–[29].

It is important to recognize that not all zone-axis patterns that can be obtained are definitive. Only those patterns with closely spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all d-spacings are less than about 0,3 nm are not definitive. A useful guideline is that the lowest angle reflections should be within the radius of the smallest ring of the gold diffraction pattern (111), and that patterns with smaller distances between reflections are usually the most definitive. It is particularly important to recognize that when ED is used to discriminate between different minerals of similar compositions, demonstration that an ED pattern is consistent with the crystal structure of a particular mineral is not proof of identity, unless the ED pattern has also been shown to be *inconsistent* with the crystal structures of the other possible minerals.

Computer programs such as XIDENT (Reference [31]) provide a convenient way to test the consistency of any given ED pattern with the crystallographic data for individual minerals. The XIDENT program is advantageous in that no knowledge of crystal orientation is required; all possible ED patterns at all orientations are calculated and compared with the observed ED pattern. If the results obtained from one ED pattern do not resolve any ambiguity in identification of a fibre, a second ED pattern obtained at a different orientation of the fibre can be examined, and the observed tilt angle between the two orientations can be compared with the theoretical angle calculated from the suspected crystal structure. In order to use the XIDENT program, five spots, closest to the centre spot, along two intersecting lines of the zone-axis pattern are selected for measurement, as illustrated in Figure F.13. The distances of these spots from the centre spot and the four angles shown provide the required data for analysis. Since the centre spot is usually very over-exposed, it does not provide a well-defined origin for these measurements. The required distances are best obtained by measuring between pairs of spots symmetrically disposed about the centre spot, preferably separated by several repeat distances.

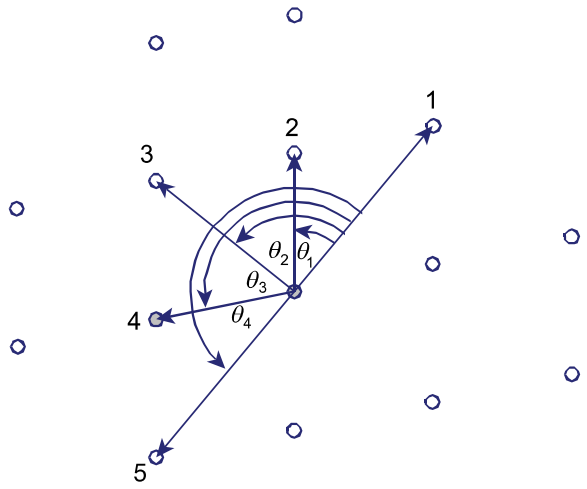


Figure F.13 — Measurement of spacings and angles in a zone axis ED pattern

Annex G
(informative)

Example of sampling record

Date:	Samples taken by:
Building and location:	

Room:		Sample identification:
Sampling location:		
Reference:	Plan No:	Position in plan:
Sketch No:		Photo No:
Sample details:		
Comments:		

Annex H
(informative)

Example of test report

Analysis of bulk materials for asbestos by ISO 22262-1

Date of analysis:			
Analyst:		Signature:	

NOTE ISO 22262-1 refers to qualitative analysis of commercial products for asbestos.

In this method, polarized light microscopy with dispersion staining is the default procedure for identification of asbestos. If the sample characteristics required the use of either of the optional electron microscope methods to identify asbestos, the method used is indicated. If accurate quantification of asbestos mass fraction in the range below approximately 5 % mass fraction is required for the purpose of determining the regulatory status of an asbestos-containing material, use the appropriate other parts of ISO 22262.

Sample	Asbestos	Estimated asbestos mass fraction	Non-asbestos fibres	Comments
Sample 20050411-1 Pipe covering Grey corrugated paper	Chrysotile	5 %–50 %	Cellulose Brucite	Sample ashed to remove interfering materials.
Sample 20050412-3 Pipe covering White fibrous material	Amosite Chrysotile	5 %–50 % 0,1 %–5 %	None	
Sample 20050412-4 Fireproofing from beam Blue fibrous material	Crocidolite	50 %–100 %	None	
Sample 20050413-1 Pipe covering Off-white fibrous material	None detected	0 %	Mineral wool	
Sample 20050413-2 Plaster White material	Tremolite	0,1 %–5 %	None	
Sample 20050413-3 Ceiling tile Grey fibrous material	Chrysotile	0,1 %–5 %	Mineral wool Cellulose	Chrysotile too fine to identify by PLM. Chrysotile identified by TEM method.

Bibliography

[1] ISO 7348:1992, *Glass containers — Manufacture — Vocabulary*

[2] ISO 10312, *Ambient air — Determination of asbestos fibres — Direct transfer transmission electron microscopy method*

[3] ISO 10934-1:2002, *Optics and optical instruments — Vocabulary for microscopy — Part 1: Light microscopy*

[4] ISO 13794:1999, *Ambient air — Determination of asbestos fibres — Indirect-transfer transmission electron microscopy method*

[5] ISO 14686:2003, *Hydrometric determinations — Pumping tests for water wells — Considerations and guidelines for design, performance and use*

[6] ISO 14952-1:2003, *Space systems — Surface cleanliness of fluid systems — Part 1: Vocabulary*

[7] ISO 14966, *Ambient air — Determination of numerical concentration of inorganic fibrous particles — Scanning electron microscopy method*

[8] AS 4964:2004, *Method for the qualitative identification of asbestos in bulk samples*

[9] EN 143, *Respiratory protective devices — Particle filters — Requirements, testing, marking*

[10] EPA/600/R-93/116:1993, *Test method, method for the determination of asbestos in bulk building materials*. Washington, DC: United States Environmental Protection Agency

[11] MDHS 77, *Asbestos in bulk materials — Sampling and identification by polarised light microscopy (PLM)*. Sudbury: HSE (UK Health and Safety Executive) Books, 1999

[12] NF X46-020:2008, *Repérage amiante — Repérage des matériaux et produits contenant de l'amiante dans les immeubles bâtis — Mission et méthodologie* [Location of asbestos — Location of materials and products containing asbestos in buildings — Mission and methodology]

[13] VDI 3866 Part 1:2000, *Determination of asbestos in technical products — Principle — Sampling and sample preparation*

[14] VDI 3866 Part 4:2002, *Determination of asbestos in technical products — Phase contrast optical microscopy method*

[15] VDI 3866 Part 5:2004, *Determination of asbestos in technical products — Scanning electron microscopy method*

[16] WAHLSTROM, E.E. *Optical crystallography*, 2nd edition. New York, NY: Wiley, 1943

[17] MCCRONE, W.C., MCCRONE, L.B., DELLY, J.G. *Polarized light microscopy*. Chicago, IL: McCrone Research Institute, 1984

[18] MCCRONE, W.C. *Asbestos identification*. Chicago, IL: McCrone Research Institute, 1987

[19] SU, S.-C. Dispersion staining: Principles, Analytical relationships and practical applications to the determination of refractive index. *Microscope* 1998, **46**, pp. 123–146.

[20] MEEKER, G.P., BERN, A.M., BROWNFIELD, I.K., LOWERS, H.A., SUTLEY, S.J., HOEFEN, T.M., VANCE, J.S. The composition and morphology of amphiboles from the Rainy Creek Complex, Near Libby, Montana. *Am. Mineral*. 2003, **88**, pp. 1955–1969

[21] EMMONS, R.C. A set of thirty immersion media. *Am. Mineral.* 1929, **14**, pp. 482–483

[22] TYLEE, B.E., DAVIES, L.S.T., ADDISON, J. Asbestos reference standards — Made available for analysts. *Ann. Occup. Hyg*. 1996, **40**, pp. 711–714

[23] LEAKE, B.E. Nomenclature of amphiboles. *Am. Mineral.* 1978, **63**, pp. 1023–1052

[24] LEAKE, B.E., WOOLLEY, A.R., ARPS, C.E.S., BIRCH, W.D., GILBERT, M.C., GRICE, J.D., et al. Nomenclature of amphiboles: Report of the Subcommittee on Amphiboles of the International Mineralogical Association Commission on new minerals and mineral names. *Mineral. Mag.* 1997, **61**, pp. 295–321.

[25] TIMBRELL, V. Characteristics of the International Union Against Cancer standard reference samples of asbestos. *Proceedings, Pneumoconiosis International Conference*, Johannesburg, 1969

[26] WENK, H.R., editor. *Electron microscopy in mineralogy*. New York, NY: Springer, 1976

[27] GARD, J.A., editor. *The electron optical investigation of clays*. London: Mineralogical Society, 1971

[28] HIRSCH, P.B., HOWIE, A., NICHOLSON, R.B., PASHLEY, D.W., WHELAN, M.J. *Electron microscopy of thin crystals*. London: Butterworths, 1965, pp. 18–23

[29] ALDERSON, R.H., HALLIDAY, J.S. Electron diffraction. In: HAY, D.H., editor. *Techniques for electron microscopy*, 2nd edition. Oxford: Blackwell Scientific, 1965, pp. 478–524.

[30] WEBBER, J.S. A simple technique for measuring asbestos layer-line spacings during TEM analysis. *Microscope* 1998, **46**, pp. 197–200.

[31] RHOADES, B.L. XIDENT — A computer technique for the direct indexing of electron diffraction spot patterns. Dept. of Mechanical Engineering, Univ. of Canterbury, Christchurch, New Zealand, 1976. (Research Report 70/76.)

ICS 13.040.20

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